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(54) PRODUCTION OF AND PRODUCTION UNIT FOR SILICON SINGLE CRYSTAL WITH FEW CRYSTAL DEFECT, AND SILICON SINGLE CRYSTAL AND SILICON WAFER PRODUCED THEREBY

(57)Abstract:
PROBLEM TO BE SOLVED: To obtain a silicon single crystal and a silicon wafer with extremely low defect density throughout the crystal by CZ process while maintaining their high productivity, along with improving the in-plane oxygen concentration distribution of the wafer.
SOLUTION: This method for producing a silicon single crystal by Czochralski(CZ) process comprises pulling up a silicon single crystal being in growth so that the contour of the solid-liquid interface in the crystal comes within ±5 mm relative to the average of the solid-liquid interface except the periphery 5 mm; wherein for the value of temperature gradient G (temperature change/ axial crystal length) [C/cm] in the proximity of the above interface from 1,420°C to 1,350°C or from the melting point of silicon to 1,400°C, in-oven temperature is controlled so that ΔG comes within 5°C/cm {ΔG=(Ge-Gc); Gc is the temperature gradient in the central portion of the crystal [°C/cm]; Ge is the temperature gradient in the peripheral portion of the crystal [°C/cm]}.

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* NOTICES *

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CLAIMS

[Claim(s)]
[Claim 1] The manufacture approach of the silicon single crystal characterized by pulling up so that the configuration of the solid-liquid interface under crystal may be set to less than **5mm to the average of a solid-liquid interface at the time of crystal growth, the silicon single crystal raised when manufacturing a silicon single crystal with the Czochrlski method removing 5mm of circumferences.

[Claim 2] [when manufacturing a silicon single crystal with the Czochrlski method] The silicon single crystal raised at the time of crystal growth the value of temperature gradient [between 1400 degrees C] G (temperature variation / crystal orientation die length) [**/cm] from the temperature gradient between 1420 degrees C and 1350 degrees C near [under crystal] the solid-liquid interface, or the melting point of silicon The manufacture approach of the silicon single crystal characterized by controlling whenever [furnace temperature] so that **G is set to cm in less than 5 degrees C /when expressed with difference **G= (germanium-Gc) of the temperature gradient Gc of a crystal center part [**/cm], and the temperature gradient germanium of a crystal circumference part [**/cm].
[Claim 3] [when manufacturing a silicon single crystal with the Czochrlski method which impressed the magnetic field] The silicon single crystal raised at the time of crystal growth the value of temperature gradient [between 1400 degrees C] G (temperature variation / crystal orientation die length) [**/cm] from the temperature gradient between 1420 degrees C and 1350 degrees C near [under crystal] the solid-liquid interface, or the melting point of silicon The manufacture approach of the silicon single crystal characterized by controlling whenever [furnace temperature] so that **G is set to cm in less than 5 degrees C /when expressed with difference **G= (germanium-Gc) of the temperature gradient Gc of a crystal center part [**/cm], and the temperature gradient germanium of a crystal circumference part [**/cm].
[Claim 4] The manufacture approach of the silicon single crystal according to claim 3 characterized by making said magnetic field impressed into a level magnetic field.
[Claim 5] The manufacture approach of the silicon single crystal according to claim 3 or 4 characterized by making reinforcement of the magnetic field to impress more than 2000G.
[Claim 6] The manufacture approach of the silicon single crystal indicated in any 1 term of claim 1 thru/or claim 5 characterized by controlling so that the die length of the crystal of the temperature region from 1300 degrees C to 1000 degrees C under said crystal is set to 8cm or less.
[Claim 7] The manufacture approach of the silicon single crystal indicated in any 1 term of claim 1 thru/or claim 6 characterized by controlling so that the time amount which passes through the temperature region from 1300 degrees C to 1000 degrees C under said crystal becomes 80 or less minutes.
[Claim 8] Pull up from the temperature gradient between 1420 degrees C and 1350 degrees C near [under said crystal] the solid-liquid interface, or the melting point of silicon with the temperature gradient G between 1400 degrees C, and a rate is adjusted. The pull-up condition of the whole crystal surface is doubled with the boundary neighborhood of a bay can C rich field and an INTASUTI sial rich field. The manufacture approach of the silicon single crystal indicated in any 1 term of claim 1 thru/or claim 7 characterized by raising the whole surface of a crystal in a neutral field with few biases of defective concentration.

[Claim 9] In the equipment which manufactures a silicon single crystal with the Czochrlski method The silicon single crystal raised at the time of crystal growth the value of temperature gradient [between 1400 degrees C] G (temperature variation / crystal orientation die length) [**/cm] from the temperature gradient between 1420 degrees C and 1350 degrees C near [under crystal] the solid-liquid interface, or the melting point of silicon The manufacturing installation of a silicon single crystal characterized by forming whenever [furnace temperature] so that **G may be set to cm in less than 5 degrees C /when expressed with difference **G= (germanium-Gc) of the temperature gradient Gc of a crystal center part [**/cm], and the temperature gradient germanium of a crystal circumference part [**/cm].
[Claim 10] In the equipment which manufactures a silicon single crystal with the Czochrlski method which impressed the magnetic field The silicon single crystal raised at the time of crystal growth the value of temperature gradient [between 1400 degrees C] G (temperature variation / crystal orientation die length) [**/cm] from the temperature gradient between 1420 degrees C and 1350 degrees C near [under crystal] the solid-liquid interface, or the melting point of silicon The manufacturing installation of a silicon single crystal characterized by forming whenever [furnace temperature] so that **G may be set to cm in less than 5 degrees C /when expressed with difference **G= (germanium-Gc) of the temperature gradient Gc of a crystal center part [**/cm], and the temperature gradient germanium of a crystal circumference part [**/cm].
[Claim 11] The manufacturing installation of the silicon single crystal indicated to claim 9 or claim 10 which arranges a solid-liquid interface heat insulator so that a silicon single crystal may be surrounded right above [of silicon melting liquid / surface-of-hot-water] in the equipment which manufactures a silicon single crystal with the Czochrlski method, and is characterized by setting to 3-5cm the clearance between the lower limits of a solid-liquid interface heat insulator and the surfaces of hot water which surrounded the silicon single crystal.
[Claim 12] The silicon single crystal manufactured by the approach indicated in any 1 term of claim 1 thru/or claim 8.
[Claim 13] The silicon single crystal manufactured by the equipment indicated in any 1 term of claim 9 thru/or claim 11.
[Claim 14] The silicon single crystal characterized by being the silicon single crystal raised by the Czochrlski method, and oxygen density distribution of a direction perpendicular to the growth direction being 5% or less.
[Claim 15] A FPD consistency is 2 100 pieces/cm. It is the following and, for size, the SEPD consistency of 10 micrometers or more is 2 ten pieces/cm. Silicon wafer characterized by being the following.
[Claim 16] The silicon wafer of claim 15 with which the field internal division cloth of an oxygen density is characterized by being 5% or less.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention has few crystal defects and oxygen density distribution is related with the silicon single crystal and silicon wafer which were manufactured by the uniform manufacture approach of a silicon single crystal, the manufacturing installation, and the list with this approach and equipment.
[0002]
[Description of the Prior Art] In recent years, the quality demand to the silicon single crystal produced with the Czochrlski method (CZ process) used as the substrate has been increasing with detailed-izing of the component accompanying high integration of a semiconductor circuit. The consistency of a defect and the reduction of size which are especially called grown-in (Grown-in) defects, such as FPD, LSTD, and COP, are demanded.
[0003] In explaining these defects, it explains being known generally about the factor which determines each concentration of the point defect of the hole mold first called the Vacancy (it may outline Vacancy and Following V) incorporated by the silicon single crystal, and the mold silicon point defect between grids called Interstitial-Si (it may outline Interstitial-Si and Following I) incorporated.
[0004] In a silicon single crystal, V fields are Vacancy, i.e., the crevice generated from lack of a silicon atom, and a field with many things like a hole. With an I region It is the thing of a field with many lumps of the rearrangement and the excessive silicon atom which are generated when a silicon atom exists too much. Between V field and an I region The neutral (it may outline Neutral and Following N) field without lack of an atom or an excess (few) will exist. And with [even if said grown-in defect (FPD, LSTD, COP) occurs when V and I are in a condition / ****** / to the last, and it has the bias of some atoms] saturation [below], it has turned out that it does not exist as a defect.
[0005] The concentration of both this point defect is decided from the pull-up rate of the crystal in a CZ process, and relation (refer to drawing 4) with the temperature gradient G near [under crystal] the solid-liquid interface. it has been accepted theory that existence of the defect of the another kind called OSF near [boundary] V field and an I region is checked (Erich Dornberger and Wilfred vonAmmon, J.Electrochem.Soc., and Vol.143 --) No.5, May 1996., T.Abe, H.Harada, J.Chikawa, Paper presented at ICDS-12 Amsterdam, August31-September3, 1982 reference.
[0006] In these, the conventional raising method is V which is the field where the idea that neither a manufacturing cost nor OSF exists to a crystal growth rate is quick. - Many manufactures in the field called as it is rich were performed, the defect in the crystal of this field was controlled for the heat history under pull-up etc., and reduction-ization was attained. For example, it is the approach of lengthening 1150-1080-degree C pass time, and FPD etc. reducing the consistency of the defect considered to be the assembly of V, and improving the oxide-film proof-pressure property which can perform evaluation near a device property. However, in the ways of controlling the heat history under pull-up (pass time of one of temperature zone regions), such as such an approach, even if it can reduce the consistency of a defect, defective size expands it, and it has turned out that the total defective whole product is not decreasing recently.
[0007] So, recently, although it increases, it pulls up for upgrading, and a rate is lowered, or the

temperature gradient near [under crystal] the solid-liquid interface is enlarged as much as possible, and a manufacturing cost is I to some or the whole surface of a crystal. - The attempt for which defects called as it is rich, such as FPD, LSTD, and COP, use the field seldom observed is being made. However, in a place which is distant from the border area of V and I also in an I-rich field by the latest research, it turned out that the defect called large SEPD considered to be the dislocation loop which the superfluous silicon between grids gathers and is formed exists. This large SEPD (henceforth L-SEPD) has a possibility of having a bad influence on the property of a device from FPD, LSTD, COP, etc. which exist in V field.

[0008] On the other hand, high integration of a semiconductor device is asking also for the homogeneous demand within a silicon wafer side, and in order that oxygen density distribution may have direct effect on the device yield produced especially, to be distributed over homogeneity is desired especially in a wafer side. Although various things, such as convection current of silicon melt, gas ambient atmosphere conditions or crystal rotation, and rotation of a crucible, can be considered, since it be cooled in the periphery of the outside of a crystal rod which grow especially, and an inside core and easy differ as a cause by which the oxygen density distribution within the silicon wafer side manufactured with the Czochrlski method get worse, it be mentioned that a crystal growth interface (solid-liquid interface) do not become Taira and others.

[0009] That is, since the inside is hard to be cooled, growth becomes the thing of a convex configuration at delay and the bottom, and in the field of the wafer which sliced and obtained this, since growth stages differ, the crystal growth interface in the Czochrlski method will usually have striation. Consequently, in a wafer side, it will have distribution according to fluctuation of the oxygen density in the crystal growth direction.

[0010] However, in the approach of pulling up and manufacturing a rod-like single crystal rod with the Czochrlski method, the fluctuation of an oxygen density based on such an interface configuration and distribution should take place inevitably, and were conventionally considered to be an unavoidable thing. Therefore, the fluctuation and distribution based on that and an interface configuration just in the difference of extent tend to improve the oxygen density distribution within a wafer side as much as possible by controlling other factors, such as said crystal rotation, when had.

[0011]

[Problem(s) to be Solved by the Invention] This invention was not made in view of such a trouble, and it aims at doubling and improving the oxygen density distribution within a field of a silicon wafer while it obtains the silicon single crystal and wafer by the CZ process in which neither a V-rich field nor an I-rich field exists and which it continues all over a crystal and is super-low defect density, maintaining the sex from Takao.

[0012]

[Means for Solving the Problem] The silicon single crystal raised when manufacturing a silicon single crystal with the Czochrlski method is the manufacture approach of a silicon single crystal that the configuration of the solid-liquid interface under crystal is characterized by pulling up invention which it was accomplished in order that this invention might attain said purpose, and was indicated to claim 1 of this invention at the time of crystal growth so that it may be set to less than **5mm to the average of a solid-liquid interface except for 5mm of circumferences.

[0013] Thus, if flattening of the crystal growth interface (solid-liquid interface) configuration is carried out so that it may be set to less than **5mm to the average of a solid-liquid interface except for 5mm of circumferences, and a crystal is pulled up, while becoming possible to pull up a crystal in the neutral region where a V-rich field with many defects and an I-rich field are not intermingled, the oxygen density distribution within a wafer side is remarkably improvable. It is because having removed 5mm of circumferences is changing rapidly and the configuration of a solid-liquid interface is not stabilized in this field.

[0014] Moreover, invention indicated to claim 2 of this invention is set when manufacturing a silicon single crystal by the CZ process. The silicon single crystal raised at the time of crystal growth the value of temperature gradient [between 1400 degrees C] G (temperature variation / crystal orientation die length) [**/cm] from the temperature gradient between 1420 degrees C and 1350 degrees C near [under crystal] the solid-liquid interface, or the melting point of silicon When expressed with difference **G= (germanium-Gc) of the temperature gradient Gc of a crystal center

part [**/cm], and the temperature gradient germanium of a crystal circumference part [**/cm], whenever [furnace temperature] was controlled so that **G was set to cm in less than 5 degrees C /.

[0015] At the time of crystal growth, thus, by the so-called adjustment of a hot zone (henceforth HZ) Namely, if whenever [furnace temperature] is controlled so that **G is set to cm in less than 5 degrees C /when expressed with difference **G= (germanium-Gc) of the temperature gradient Gc of a crystal center part [**/cm], and the temperature gradient germanium of a crystal circumference part [**/cm] Since a pull-up only in N field which exists between a V-rich field and an I-rich field is enabled and the pull-up rate can be decided In the neutral region where a V-rich field with many defects and an I-rich field are not intermingled, it continues all over a crystal, and the silicon single crystal and wafer by the CZ process which is super-low defect density can be manufactured, maintaining the sex from Takao by the stable state. And by carrying out difference **G of a temperature gradient in less than 5 degrees C/cm in this way, except for 5mm of circumferences, flattening of the solid-liquid interface configuration at the time of crystal training can be carried out to less than **5mm to the average of a solid-liquid interface, and oxygen density distribution can be improved. In this case, although the temperature gradient between 1400 degrees C may be used from the melting point of silicon, using the temperature gradient between 1420 degrees C and 1350 degrees C as temperature gradient [near / under crystal / the solid-liquid interface] G (temperature variation / crystal orientation die length) [**/cm], control with more exact using the temperature gradient between 1400 degrees C from the melting point of silicon can be carried out.

[0016] Next, invention indicated to claim 3 of this invention is set when manufacturing a silicon single crystal with the Czochrlski method which impressed the magnetic field. The silicon single crystal raised at the time of crystal growth the value of temperature gradient [between 1400 degrees C] G (temperature variation / crystal orientation die length) [**/cm] from the temperature gradient between 1420 degrees C and 1350 degrees C near [under crystal] the solid-liquid interface, or the melting point of silicon When expressed with difference **G= (germanium-Gc) of the temperature gradient Gc of a crystal center part [**/cm], and the temperature gradient germanium of a crystal circumference part [**/cm], it is the manufacture approach of the silicon single crystal characterized by controlling whenever [furnace temperature] so that **G is set to cm in less than 5 degrees C /.

[0017] Thus, if **G pulls up a crystal in the Czochrlski method which impressed the magnetic field as it is set to cm in less than 5 degrees C /, since N field will become [breadth and its control range] large, the silicon single crystal and silicon wafer which do not almost have a crystal defect more simply can be manufactured.

[0018] In this case, as are indicated to claim 4, and the magnetic field impressed is made into a level magnetic field and indicated to claim 5, it is desirable to make reinforcement of the magnetic field to impress more than 2000G. While controlling the convection current of silicon melt and extending N field, in order to carry out flattening of the solid-liquid interface configuration, the level magnetic field is more desirable than a vertical magnetic field or a cusp field, and it is because there is little magnetic field impression effectiveness in less than [2000G].

[0019] And in this invention, it is desirable to control so that the crystal die length of the temperature region from 1300 degrees C to 1000 degrees C under crystal is set to 8cm or less (claim 6), and to control so that the time amount which passes through the temperature region from 1300 degrees C to 1000 degrees C under crystal becomes 80 or less minutes (claim 7).

[0020] By [which specified the temperature gradient or pull-up rate of a solid-state crystal part above the solid-liquid interface under said crystal] pulling up, being conditions and carrying out like this, the absolute value of a temperature gradient G itself becomes large, and it can pull up this in a neutral field also with a high pull-up rate.

[0021] When the crystal die length of the temperature region from 1300 degrees C to 1000 degrees C under crystal exceeds 8cm, the absolute value of a temperature gradient G becomes small, and it continues all over a crystal only at an extremely late raising rate, and it becomes impossible to obtain the silicon single crystal and wafer which are super-low defect density. if the absolute value of a temperature gradient G tends to become small, it is going to continue all over a crystal and it is going to obtain the silicon single crystal and wafer which are super-low defect density, even when similarly the time amount which passes through the temperature region from 1300 degrees C to 1000 degrees

C under crystal exceeds 80 minutes -- a raising rate -- late -- not carrying out -- it does not obtain but it becomes difficult to maintain the sex from Takao by the stable state.
[0022] Moreover, invention indicated to claim 8 of this invention pulls up from the temperature gradient between 1420 degrees C and 1350 degrees C near [under crystal] the solid-liquid interface, or the melting point of silicon with the temperature gradient G between 1400 degrees C, and adjusts a rate. The pull-up condition of the whole crystal surface is doubled with the boundary neighborhood of V and a rich field, and an I and a rich field, and it is made to perform the whole surface of a crystal in a neutral (N, neutrality) field with few biases of defective concentration.
[0023] When the crystal was pulled up by the approach of not taking into consideration the concept of the conventional N field at all, difference **G= (germanium-Gc) of a temperature gradient was not able to become large, and was not able to make only N field in the crystal face, but as described above, while controlling to **G=(germanium-Gc) <=5[**/cm], N field came be made on all over the crystal by adjusting a pull-up rate. By carrying out like this, there is little any generating of the defect of V and I, defective concentration is low on the whole surface of a crystal, a neutral field with few the bias is obtained, and the stable quality is secured.
[0024] Next, invention indicated to claim 9 of this invention is set to the equipment which manufactures a silicon single crystal with the Czochrlski method. The silicon single crystal raised at the time of crystal growth the value of temperature gradient [between 1400 degrees C] G (temperature variation / crystal orientation die length) [**/cm] from the temperature gradient between 1420 degrees C and 1350 degrees C near [under crystal] the solid-liquid interface, or the melting point of silicon When expressed with difference **G= (germanium-Gc) of the temperature gradient Gc of a crystal center part [**/cm], and the temperature gradient germanium of a crystal circumference part [**/cm], it is the manufacturing installation of a silicon single crystal characterized by forming whenever [furnace temperature] so that **G may be set to cm in less than 5 degrees C /.
[0025] Moreover, invention indicated to claim 10 of this invention In the equipment which manufactures a silicon single crystal with the Czochrlski method which impressed the magnetic field The silicon single crystal raised at the time of crystal growth the value of temperature gradient [between 1400 degrees C] G (temperature variation / crystal orientation die length) [**/cm] from the temperature gradient between 1420 degrees C and 1350 degrees C near [under crystal] the solid-liquid interface, or the melting point of silicon When expressed with difference **G= (germanium-Gc) of the temperature gradient Gc of a crystal center part [**/cm], and the temperature gradient germanium of a crystal circumference part [**/cm], it is the manufacturing installation of a silicon single crystal characterized by forming whenever [furnace temperature] so that **G may be set to cm in less than 5 degrees C /.
[0026] And invention indicated to claim 11 of this invention is equipment according to claim 9 or 10, and is a manufacturing installation of a silicon single crystal which arranges a solid-liquid interface heat insulator so that a silicon single crystal may be surrounded right above [of silicon melting liquid / surface-of-hot-water], and is characterized by setting to 3-5cm the clearance between the lower limits of a solid-liquid interface heat insulator and the surfaces of hot water which surrounded the silicon single crystal in the equipment which manufactures a silicon single crystal with the Czochrlski method.
[0027] Thus, if **G is the equipment set to cm in less than 5 degrees C /, especially equipment which impressed the magnetic field, it will become possible to pull up a crystal only in N field without a crystal defect. For example, if it is made the structure which prepared the 3-5cm clearance and installed the solid-liquid interface heat insulator lower limit which surround HZ from the surface of hot water The radiant heat of a heater hits a solid-liquid interface enough, and it becomes possible to form the manufacturing installation of the silicon single crystal which can control [cm] difference **G= (germanium-Gc) of the temperature gradient near the solid-liquid interface in 5 degrees C /or less. As a result, it is stabilized and the silicon single crystal and silicon wafer of very little quality of a crystal defect can be manufactured.
[0028] The thermal radiation from a heater etc. becomes inadequate, when the clearance between this solid-liquid interface heat insulator lower limit and surface of hot water is less than 3cm, and it becomes the value to which difference **G= (germanium-Gc) of the temperature gradient near the

solid-liquid interface exceeds cm in 5 degrees C /as a result, and it continues all over a crystal and it becomes impossible to be the neutral region where a V-rich field with many defects and an I-rich field are not intermingled, and to make the silicon single crystal and wafer which are super-low defect density. On the other hand, when the clearance between this solid-liquid interface heat insulator lower limit and surface of hot water exceeds 5cm, in order for the value of the temperature gradient G itself to become small, to continue all over a crystal inevitably and to obtain the silicon single crystal and wafer which are super-low defect density, it will be necessary to pull up and to make a rate extremely late, and, now, it is difficult [it] to maintain the sex from Takao by the stable state.

[0029] Furthermore, invention indicated to claim 12 of this invention and claim 13 is the silicon single crystal manufactured by the equipment indicated in any 1 term of the approach indicated in any 1 term of said claim 1 thru/or claim 8 or claim 9 thru/or claim 11.

[0030] Thus, while there are very few crystal defects, such as FPD, LSTD, COP, and L-SEPD, since a crystal can be stably pulled up in N field if a silicon single crystal is manufactured with the equipment indicated in any 1 term of the approach indicated in any 1 term of said claim 1 thru/or claim 8 and claim 9 thru/or claim 11, the silicon single crystal which has improved the oxygen density distribution within a wafer side can be manufactured.

[0031] Next, it is the silicon single crystal characterized by for invention indicated to claim 14 of this invention being the silicon single crystal raised by the Czochrlski method, and oxygen density distribution of a direction perpendicular to the growth direction being 5% or less. Thus, in this invention, since **G is carried out in less than 5 degrees C/cm and flattening of the solid-liquid interface configuration is carried out, a silicon single crystal with uniform oxygen density distribution can be obtained. Especially, in this invention, most one single crystal rod will become uniform in a direction (wafer side inboard after a slice) perpendicular to the growth direction.

[0032] And for invention indicated to claim 15 of this invention, a FPD consistency is 2 100 pieces/cm. It is the following and, for size, the SEPD consistency of 10 micrometers or more is 2 ten pieces/cm. It is the silicon wafer characterized by being the following.

[0033] Thus, the silicon wafer produced from the silicon single crystal manufactured by this invention has very few grown-in defects, such as FPD, LSTD, COP, and L-SEPD, and they can use it as a very useful silicon wafer.

[0034] And as indicated to claim 16, the silicon wafer of this invention can also make the field internal division cloth of an oxygen density 5% or less while having few crystal defects. Although the method of a display here of the field internal division cloth of an oxygen density has various things, in this invention, the maximum measured in the field, the value [or] which broke the difference of the minimum value by maximum, the maximum measured in the field, or the value which broke the difference of the minimum value by the average can be made into 5% or less.

[0035] Hereafter, although explained to a detail per this invention, this invention is not limited to these. In advance of explanation, lessons is taken from each vocabulary, and it explains beforehand.

1) K2 Cr 2O7 after cutting down a wafer from the silicon single crystal rod after growth and etching and removing a surface distortion layer with the mixed liquor of fluoric acid and a nitric acid in FPD (Flow Pattern Defect) A pit and a ripple pattern arise by etching a front face with the mixed liquor of fluoric acid and water (Secco etching). This ripple pattern is called FPD, and the defects of oxide-film pressure-proofing increase in number, so that the FPD consistency within a wafer side is high (refer to JP,4-192345,A).

[0036] 2) When the same Secco etching as FPD is performed, call SEPD (Secco Etch Pit Defect) a thing without FPD, a call, and a flow pattern for the thing accompanied by a flow pattern (flow pattern) with SEPD. When it is thought in this that large SEPD 10 micrometers or more originates in a rearrangement cluster and a rearrangement cluster exists in a device, a current leaks through this rearrangement and it stops achieving the function as a P-N junction.

[0037] 3) Cut down a wafer from the silicon single crystal rod after growth, and carry out cleavage of the wafer to LSTD (Laser Scattering TomographyDefect) after etching and removing a surface distortion layer with the mixed liquor of fluoric acid and a nitric acid. Incidence of the infrared light can be carried out from this cleavage plane, and the defect scattering light which exists in a wafer can be detected by detecting the light which came out from the wafer front face. About the scatterer

observed here, it is a society etc., there is already a report, and it is regarded as the oxygen sludge (J. J.A.P. Vol.32, P3679, 1993 reference). Moreover, the result that it is the void (hole) of octahedron is also reported by the latest research.

[0038] 4) the defect which becomes the cause of degrading oxide film pressure-proofing of the core of a wafer, with COP (Crystal Originated Particle) -- it is -- Secco -- by ammonia hydrogen-peroxide-solution washing (washing by the mixed liquor of NH4 OH:H2 O2:H2 O=1:1-2:5-7), the defect set to FPD if dirty works as a selection etching solution, and is set to COP. The diameter of this pit is investigated with light scattering measurement by 1 micrometer or less.

[0039] the place which this invention persons investigated in the detail about the boundary neighborhood of V field and an I region about the silicon single crystal growth by the CZ process -- **** of this boundary neighborhood -- the narrow field had few FPD(s), LSTD(s), and COP remarkably, and it discovered that there was a neutral field where big SEPD does not exist, either.

[0040] Then, when this neutral field could be extended all over the wafer, it was conceived that a point defect could be reduced sharply. as shown in drawing 4 , the main factors which determine concentration distribution of the point defect within a field in said growth (pull-up) rate and relation of a temperature gradient which were carried out since a pull-up rate is about 1 law in the wafer side of a crystal are temperature gradients. That is, in the wafer side, on the problem, when that a difference is in the temperature gradient of shaft orientations could reduce this difference, it found out that the concentration difference of the point defect within a wafer side could also be reduced.

[0041] And when abolishing the difference of the temperature gradient of shaft orientations in the wafer side in this way, it turned out that flattening of the solid-liquid interface of a pull-up silicon single crystal can be carried out, and an improvement of the oxygen density distribution within a wafer side can also be performed.

[0042] Here, when the difference of the temperature gradient Gc of the crystal center section and the temperature gradient germanium of a crystal circumference part as shown in drawing 5 in the case of an approach to pull up usual was investigated, those with at least 15 degree C/cm, and when the absolute value of a temperature gradient G was enlarged especially, it was checked for (germanium-Gc) by (germanium-Gc) breadth and that there is occasionally a difference also cm in 40 degrees C /.

[0043] Thus, if a difference is in the temperature gradient of the crystal center section and a circumference part, like drawing 5 , the configuration of the solid-liquid interface (crystal growth interface) 4 will not become, but flatness will turn in a convex configuration up. And if there is difference **G of a temperature gradient also cm in 15 degrees C /as mentioned above, the solid-liquid interface of a core will come to exceed **5mm to the average of a solid-liquid interface except for 5mm of circumferences. Therefore, in the field of the wafer which was sliced and was obtained from the crystal rod by such the conventional Czochrlski method, it will have the striation resulting from growth stages differing, and will have distribution according to fluctuation of the oxygen density in the crystal growth direction.

[0044] The approach of reducing the difference of a temperature gradient G then, for example The comprehensive heat transfer analysis software (F. 33 Dupret, P.Nicodeme, Y.Ryckmans, P.Wouters, and M.J.Crochet, Int.J.Heat Mass Transfer, 1849 (1990)) called FEMAG When it is used and investigates wholeheartedly, as shown in drawing 6 , the range of 1400 degrees C is kept warm with a heat insulator from the melting point of 1420-1350 degrees C which is the pyrosphere of a crystal, or silicon. Moreover, near the solid-liquid interface, the radiant heat from melt was applied directly, and, on the other hand, this showed that what is necessary was just to cool a low-temperature part as much as possible.

[0045] If a 3-5cm clearance is prepared and installed between the lower limits of a solid-liquid interface heat insulator and the surfaces of hot water which have specifically arranged the solid-liquid interface heat insulator so that a silicon single crystal may be surrounded right above [of silicon melting liquid / surface-of-hot-water], and surrounded the silicon single crystal The radiant heat of a heater hit the solid-liquid interface enough, the heat distribution from which difference **G= (germanium-Gc) of a temperature gradient like drawing 3 which showed the relation between a crystal growth rate and a temperature gradient is set to cm in 5 degrees C /or less was formed, and it became clear that a hot zone with few V and I defects exists.

[0046] In an approach to pull up a silicon single crystal Therefore, the above temperature gradients, Namely, the value of temperature gradient [between 1400 degrees C] G (temperature variation / crystal orientation die length) [**/cm] from the temperature gradient between 1420 degrees C (melting point of silicon), and 1350 degrees C near [under crystal] the solid-liquid interface, or the melting point of silicon If HZ adjusts whenever [furnace temperature] so that **G may be set to cm in less than 5 degrees C /when expressed with difference **G= (germanium-Gc) of the temperature gradient Gc of a crystal center part [**/cm], and the temperature gradient germanium of a crystal circumference part [**/cm] The pull-up rate of the crystal of N field which exists between V rich field and I rich field can be decided, the whole wafer surface becomes neutral, and most point defects are no longer seen (R> drawing 4 4 reference).
[0047] Also in **G<=5 degree-C/cm HZ, the single crystal of a whole surface N rich field is obtained so that a growth rate may be seen in the middle of drawing 4 by ** or becoming the crystal of V rich field if it passes, becoming the crystal of I rich field so that the lower part may see if too late, and choosing a suitable growth rate, so that it may see in the upper part of drawing 4 .
[0048] When control of this temperature gradient and the relation of a crystalline region are explained more concretely, as shown in drawing 7 , in the conventional CZ process For example, if it is **G=germanium-Gc=20 degree-C/cm HZ when Gc=30 degree-C/cm and germanium=50 degree-C/cm, for example, it raises with a comparatively early growth rate in the location of A of drawing 7 If the crystalline region in this condition is seen on the single crystal cross section as shown in drawing 9 (a), a core and a periphery are V field with many [respectively] crystal defects, and an I region, and N field with few defects exists in these pars intermedia in the shape of a circular ring.
[0049] On the other hand, by the approach of this invention, so that it may see in the location of B of drawing 7 , when Gc=35 degree-C/cm and germanium=40 degree-C/cm, it is **G=germanium-Gc=5 degree-C/cm HZ and was raising with the comparatively late growth rate, and as shown in drawing 9 (b), when the crystalline region in this condition was seen on the single crystal cross section, the whole surface became N field with few defects, for example.
[0050] However, in this condition, if the absolute value of a temperature gradient G (Gc and germanium) is set to enlarged HZ, maintaining difference **G=germanium-Gc<=5 degree-C/cm of a temperature gradient from the location of C to the location of D as shown in drawing 8 since the growth rate was slow, a whole surface N field can be attained now with an early growth rate, and high productivity can be maintained.
[0051] Moreover, when this phenomenon was seen by the temperature gradient of the already crystallized part which is located above a solid-liquid interface, it turned out that it can attain by controlling so that the crystal die length of the temperature region from 1300 degrees C to 1000 degrees C is set to 8cm or less. Since the absolute value of a temperature gradient will become small if this temperature region spreads from 8cm in a crystal, a late pull-up rate must be chosen and it results in worsening productive efficiency.
[0052] Furthermore, it is required to control so that the time amount which passes through the temperature region from 1300 degrees C in the part which pulled up this phenomenon and was already crystallized seen from the field of a rate to 1000 degrees C becomes 80 or less minutes. In order for the absolute value of a temperature gradient to become small and to obtain the crystal in N field also by annealing which exceeds 80 minutes, it will pull up, a rate must be made late and productive efficiency will fall.
[0053] And if **G is pulled up as less than 5 degrees C/cm in this way, a solid-liquid interface configuration is set to less than **5mm to the average of a solid-liquid interface except for 5mm of circumferences, and while being set to less than **2.5mm and becoming easy to grow up the crystal of N field, the field internal division cloth of an oxygen density will also be improved especially.
[0054] As mentioned above, although the pull-up conditions in the neutral field of this invention have been clarified If these are summarized, will pull up from the temperature gradient between 1420 degrees C and 1350 degrees C near [under crystal] the solid-liquid interface, or the melting point of silicon with the temperature gradient G between 1400 degrees C, and a rate will be adjusted. I hear that the pull-up condition of the whole crystal surface is doubled with the boundary neighborhood of the bay can (C V) rich field and the INTASUTI (sial I) rich field, and it is made to perform the whole surface of a crystal in a neutral field with few biases of defective concentration,

and it is. And the field internal division cloth of an oxygen density is also remarkably improved by this.
[0055] In addition, in order to get to know the pull-up rate for pulling up the whole surface of the crystal at the time of carrying out **G in less than 5 degrees C/cm, and making the absolute value of a temperature gradient into a request value in N field For example, it is made to fall even to the growth rate which reduces a growth rate gradually, pulling up a single crystal rod at high speed so that **G may become in less than 5 degrees C/cm, and a V-rich crystal's being made to be made, and maintaining **G in less than 5 degrees C/cm after that, and can finally do an I-rich crystal. And it will be V, if the made single crystal rod is cut to a lengthwise direction and a crystal defect is investigated. - It is I that it is rich. - While it is rich, the growth rate of existing N field can be known.

[0056] In this case, if a crystal is pulled up maintaining [cm] **G in less than 5 degrees C /with the Czochrlski method (MCZ law) which impresses a magnetic field to silicon melt, the range of a pull-up rate where the above-mentioned N field turns into breadth and N field will spread, and it will become possible to pull up a single crystal as an N field easily.
[0057]
[Embodiment of the Invention] Hereafter, the operation gestalt of this invention is explained to a detail, referring to a drawing. First, drawing 1 explains the example of a configuration of the crystal-pulling equipment by the CZ process of this invention. As shown in drawing 1 , this crystal-pulling equipment 30 The pull-up room 31, the crucible 32 prepared all over the pull-up room 31, and the heater 34 arranged around a crucible 32, It has the reel style (not shown) which rotates or rolls round the crucible maintenance shaft 33 made to rotate a crucible 32 and its rolling mechanism (not shown), the seed chuck 6 holding the seed crystal 5 of silicon, the cable 7 that pulls up a seed chuck 6, and a cable 7, and is constituted. A quartz crucible is prepared in the side in which a crucible 32 holds the silicon melt (molten bath) 2 of the inside, and the graphite crucible is prepared in the outside. Moreover, the heat insulator 35 is arranged around [outside] the heater 34.
[0058] Moreover, in order to set up the manufacture conditions in connection with the manufacture approach of this invention, the annular solid-liquid interface heat insulator 8 is formed in the periphery of the solid-liquid interface of a crystal, and the up surrounding heat insulator 9 is arranged on it. This solid-liquid interface heat insulator 8 forms the 3-5cm clearance 10 between that lower limit and surface of hot water of silicon melt 2, and is installed in it. The up surrounding heat insulator 9 may not be used depending on conditions. Furthermore, coolant gas is sprayed or the tubed cooling system 36 which interrupts radiant heat and cools a single crystal is formed.
[0059] Independently, recently, the convection current of melt is controlled by installing the magnet 38 which consists of usual state conduction or a superconduction coil in the horizontal outside of the pull-up room 31, and impressing magnetic fields, such as a horizontal direction or a perpendicular direction, to silicon melt 2 so that drawing 12 may see, and the so-called MCZ method for measuring the stable growth of a single crystal is used in many cases. The direction of the magnetic field impressed to melt can be easily changed by magnetic arrangement. For example, if one coil is arranged so that the pull-up room 31 may be surrounded horizontally, a vertical magnetic field (vertical magnetic field) will be impressed to melt, and if opposite arrangement of the two coils is carried out on the horizontal outside of the pull-up room 31, a horizontal magnetic field (horizontal magnetic field) will be impressed to melt. And also in this invention, if this MCZ method is used as mentioned above, a breadth control range can spread and N field can obtain N crystal easily.
[0060] Next, the single-crystal-growth approach by the crystal-pulling equipment 30 of above-mentioned drawing 1 is explained. First, within a crucible 32, the high grade polycrystal raw material of silicon is heated more than the melting point (about 1420 degrees C), and is dissolved. Next, the tip of a seed crystal 5 is made contacted or immersed in the surface abbreviation core of a molten bath 2 by beginning to roll a cable 7. Then, while rotating the crucible maintenance shaft 33 in the proper direction, single crystal growth is started by rolling round rotating a cable 7 and pulling up a seed crystal 5. Henceforth, the single crystal rod 1 of an approximate circle column configuration can be obtained by adjusting a raising rate and temperature appropriately.
[0061] In this case, in this invention, in order to attain the purpose of this invention, the following facilities were performed. First, as shown in drawing 6 (partial expansion explanatory view of

drawing 1 R> 1), in the periphery space of the liquefied part in the single crystal 1 on the surface of hot water of the pull-up room 31, the temperature of the crystal near the surface of hot water formed the annular solid-liquid interface heat insulator 8 in the temperature region from 1420 degrees C to 1350 degrees C (from the melting point of silicon to or 1400 degrees C), and the so-called HZ, and arranges the up surrounding heat insulator 9 on it. This solid-liquid interface heat insulator 8 forms the 3-5cm clearance 10 between that lower limit and surface of hot water 3 of silicon melt 2, and is installed in it. The up surrounding heat insulator 9 may not be used depending on conditions. Furthermore, it may consider as the structure which should form the equipment 36 which cools a crystal, for example, a cooling system, in the upper part of this heat insulator, should spray coolant gas on this from the upper part, should cool the crystal, and attached the radiant heat reflecting plate in the cylinder lower part.

[0062] Thus, by establishing a predetermined clearance in the location of the right above of an oil level, arranging a heat insulator, and considering as the structure which formed further the equipment which cools a crystal in the upper part of this heat insulator Since a heat insulation effect is acquired by radiant heat near the crystal growth interface and the radiant heat from a heater etc. can be cut in the upper part of a crystal The temperature gradient germanium of a crystal periphery became small, the difference with the temperature gradient Gc of the crystal center section also became small, and the solid-liquid interface 4 also became a flat and became possible [pulling up a crystal in N field with few biases of defective concentration on the whole surface of a crystal]. An air-cooling duct, a water-cooled coil, etc. which surround the perimeter of a crystal are formed, and you may make it secure a desired temperature gradient independently [said tubed cooling system 36] as a cooling system of this crystal.

[0063] Moreover, it is desirable to arrange a magnet 38 on the outside of the pull-up room 31 like drawing 12 in this invention. In this case, the magnetic field impressed is made into a level magnetic field, and reinforcement of the magnetic field to impress is more preferably made more than 3000G more than 2000G. By impressing a magnetic field, the convection current of silicon melt is controlled and N field under crystal spreads. Moreover, in order to carry out flattening of the solid-liquid interface configuration, the level magnetic field is more desirable than a vertical magnetic field or a cusp field, and it is because there is little magnetic field impression effectiveness in less than [2000G].

[0064] And an improvement of quality can be aimed at in this invention, without reducing the productivity of a single crystal, since there is not no need of reducing a raising rate extremely like before and it is not necessary to anneal a fixed temperature region and. And the field internal division cloth of an oxygen density is also improved, if the MCZ method is used, a control range will become large and manufacture of a low defective crystal can be performed certainly.

[0065] Conventional equipment was shown in drawing 2 for drawing 1 , the crystal-pulling equipment of this invention of drawing 12 , and a comparison. Although it is the same as the raising equipment of this invention about fundamental structure, the solid-liquid interface heat insulator 8, the up surrounding heat insulator 9, or cooling system 36 of this invention are not equipped.

[0066] As mentioned above, since the approach explained to the detail and the silicon single crystal manufactured by equipment can pull up a crystal stably in N field, it can be made into very few things of crystal defects, such as FPD, LSTD, COP, and L-SEPD. moreover, a single crystal -- the whole can be covered mostly and oxygen density distribution of a direction perpendicular to the crystal growth direction can be made into 5% or less.

[0067] The silicon wafer produced from such a silicon single crystal It sets all over the and a FPD consistency is 2 100 pieces/cm. It is the following and, for size, the SEPD consistency of 10 micrometers or more is 2 ten pieces/cm. By becoming the silicon wafer which is the following and performing oxygen precipitation heat treatment When X-ray observation is carried out after oxygen precipitation heat treatment to a silicon wafer with an oxygen density in which oxygen deposits, the contrast of the deposit within a field is fixed and a wafer with few striation rings is obtained. That is, since the growth interface is flat, the homogeneity within a wafer side is good and especially the oxygen density distribution within a field becomes 5% or less.

[0068]

[Example] Hereafter, the example of this invention is shown.

(Example 1) With the raising equipment 30 shown in drawing 1 , 60kg of raw material polycrystalline silicon was charged to the 20 inch quartz crucible, the diameter of 6 inches and the silicon single crystal rod of bearing <100> were changed to 1.0 - 0.4 mm/min, and the average raising rate was pulled up (body die length of about 85cm of a single crystal rod). It was made into 4cm space from 1420 degrees C and the surface of hot water up to the lower limit of an annular solid-liquid interface heat insulator, on it, the water temperature of silicon melt has arranged the annular solid-liquid interface heat insulator of 10cm height, it adjusted the crucible maintenance shaft, set the height from the surface of hot water to pull-up room head lining as 30cm, and arranged the up surrounding heat insulator.
[0069] The temperature gradient between 1400 degrees C was set as **/**/germanium=45.0[cm] Gc=42.0[cm] **G=(germanium-Gc) =3.0[**/cm] from the melting point of the silicon near the solid-liquid interface of a crystal. The mirror plane wafer of a silicon single crystal was produced by cutting down a wafer and performing mirror plane processing from the single crystal rod obtained here. In this way, measurement of said FPD and a L-SEPD defect was performed about the mirror plane wafer of the made silicon single crystal. A pull-up rate and the relation of a defective measurement result were shown in Table 1. Moreover, the silicon single crystal rod completely grown up similarly was made into vertical division, and drawing 10 (a) looked at the situation of change of the crystal defect in the growth direction.
[0070]
[Table 1]

引上げ速度 (mm/min)	温度勾配の差 G=Ge−Gc (℃/cm)	領　域	FPD (/cm²)	L−SEPD (/cm²)
0.8	3.6	Vリッチ領域	約1000	—
0.55	3.6	N 領　域	≦20	—
0.5	3.6	Iリッチ領域	≧20	≧10

[0071] if a pull-up rate does not suit even if there is no difference in the concentration of the point defect by which **G is incorporated in the field of a crystal as less than 5 degrees C/cm so that the result of Table 1 may show -- the result -- V -- rich -- I -- since it became rich, it searched for a pull-up rate which suits N field exactly (refer to drawing 4). consequently, the case where it pulls up at the pull-up rate of 0.55 mm/min -- the whole surface -- the neutral silicon wafer was producible. And the crystal growth interface configuration 43 is flat, and is uniform so that drawing 10 (a) may see. [of oxygen density distribution] However, the range of the N field 39 between the V-rich field 40 and the I-rich field 41 is comparatively narrow, and in order to grow up the whole crystal as this N field 39, it needs highly precise control. In addition, the OSF field 42 is seen between N fields.
[0072] (Example 2) Next, the crystal was pulled up with the raising equipment shown in drawing 12 , impressing the level magnetic field of 3000G to melt. It considered as the same conditions as an example 1 except having impressed the magnetic field. That is, 60kg of raw material polycrystalline silicon was charged to the 20 inch quartz crucible, the diameter of 6 inches and the silicon single crystal rod of bearing <100> were changed to 1.0 - 0.4 mm/min, and the average raising rate was pulled up. It was made into 4cm space from 1420 degrees C and the surface of hot water up to the lower limit of an annular solid-liquid interface heat insulator, on it, the water temperature of silicon

melt has arranged the annular solid-liquid interface heat insulator of 10cm height, it adjusted the crucible maintenance shaft, set the height from the surface of hot water to pull-up room head lining as 30cm, and arranged the up surrounding heat insulator.
[0073] The temperature gradient between 1400 degrees C was set as **/**/germanium=45.0[cm] Gc=42.0[cm] **G=(germanium-Gc) =3.0[**/cm] from the melting point of the silicon near the solid-liquid interface of a crystal. The single crystal rod obtained here was made into vertical division, and drawing 10 (b) looked at the situation of change of the crystal defect in the growth direction.
[0074] the case where it usually pulls up at the pull-up rate near 0.55 - 0.58 mm/min like a CZ process when drawing 10 (b) was seen -- the whole surface -- it turns out that a neutral silicon wafer is producible. Moreover, the crystal growth interface configuration 43 is also flat, and oxygen density distribution will also become uniform.
[0075] And it turns out that it becomes what is easy to serve as N field in the same wafer side altogether to differ from the usual CZ process of drawing 10 (a) greatly when the control range of breadth and a pull-up rate carries out flattening also of the boundary of spreading remarkably and a V-rich field, N field, and an I-rich field, slices this and N field considers as a wafer very much.
[0076] Thus, although the detail of the reason which changes remarkably signs that a crystal defect occurs is unknown for the moment if a magnetic field is impressed Since the convection current of melt is stabilized by impressing a magnetic field, the temperature gradient in melt changes and the amount of incorporation of the defect of a under [a crystal] itself changes, It is thought that it is because it became the ideal temperature gradient which the temperature gradient under crystal near the growth interface also influences and stabilizes the effect of change of the temperature gradient in melt, and does not have a crystal defect.
[0077] (An example 3, example of a comparison) Next, the solid-liquid interface configuration investigated the effect which it has had on the oxygen density within a wafer side. The diameter of 8 inches and the silicon single crystal rod of bearing <100> were pulled up having charged 60kg of raw material polycrystalline silicon to the 20 inch quartz crucible, and impressing the magnetic field of 3000G with the raising equipment shown in drawing 12 . It was made into 4cm space from 1420 degrees C and the surface of hot water up to the lower limit of an annular solid-liquid interface heat insulator, on it, the water temperature of silicon melt has arranged the annular solid-liquid interface heat insulator of 10cm height, it adjusted the crucible maintenance shaft, set the height from the surface of hot water to pull-up room head lining as 30cm, and arranged the up surrounding heat insulator.
[0078] The temperature gradient between 1400 degrees C was set as **/**/germanium=32.6[cm] Gc=30.5[cm] **G=(germanium-Gc) =2.1[**/cm] from the melting point of the silicon near the solid-liquid interface of a crystal. The obtained silicon single crystal rod was made into vertical division, and drawing 11 R> 1 (b) looked at the situation of change of the oxygen density in the growth direction in the crystal center section and a periphery (example 3). In this case, most configurations of the solid-liquid interface under crystal were flatness in the convex configuration of less than **2mm to the average of a solid-liquid interface except for 5mm of circumferences.
[0079] The silicon single crystal rod with a diameter of 8 inches was made to raise, removing a solid-liquid interface heat insulator and an up surrounding heat insulator, making other conditions the same as that of the above on the other hand, and impressing the magnetic field of 3000G. At this time, the temperature gradient near the solid-liquid interface of a crystal was set as **/**/germanium=63.5[cm] Gc=30.4[cm] **G=(germanium-Gc) =33.1[**/cm]. The obtained silicon single crystal rod was made into vertical division, and drawing 11 (a) looked at the situation of change of the oxygen density in the growth direction in the crystal center section and a periphery (example of a comparison). In this case, the configuration of the solid-liquid interface under crystal was **10mm in convex configuration to the average of a solid-liquid interface except for 5mm of circumferences.
[0080] When drawing 11 is seen, it turns out that there is a difference at the core and periphery of a crystal with a big oxygen density, and the oxygen density differs in (a) greatly in the growth direction. And it turns out that fluctuation of the growth direction of the oxygen density in a core and fluctuation of the oxygen density in a periphery are carrying out fluctuation almost same at about 12-

20mm phase contrast. This makes the convex configuration of a crystal growth interface reflect as it is.
[0081] on the other hand -- (b) -- an oxygen density -- the core and periphery of a crystal -- about -- I am doing one and it turns out that fluctuation of the growth direction of the oxygen density in a core and fluctuation of the oxygen density in a periphery are very well in agreement at about 0-3mm phase contrast. Oxygen density distribution of the direction where dispersion in the growth direction of this silicon single crystal is perpendicular to the growth direction of a certain thing is very good, and when this is sliced and it considers as a wafer, distribution of an oxygen density will become very good in a field. That flattening of the crystal growth interface was carried out makes this reflect.

[0082] In addition, this invention is not limited to the above-mentioned operation gestalt. The above-mentioned operation gestalt is instantiation, and no matter it may be what thing which has the same configuration substantially with the technical thought indicated by the claim of this invention, and does the same operation effectiveness so, it is included by the technical range of this invention.
[0083] For example, in the above-mentioned operation gestalt, when a diameter 6 and a 8 inches silicon single crystal were raised, the example was given and explained per, but this invention is applicable also to the diameter of 8-16 inches, or the silicon single crystal beyond it, if it adjusts at the pull-up rate which is not limited to this, agrees to N field, and makes small the difference of the temperature gradient of the crystal center section near the solid-liquid interface of a crystal, and a periphery. moreover, MCZ of others which are not restricted when impressing a level magnetic field to silicon melt, and impress a vertical magnetic field, a cusp field, etc. also when impressing a magnetic field by this invention -- it cannot be overemphasized that it is applicable also to law.
[0084]
[Effect of the Invention] it explained above -- as -- this invention -- a CZ process and MCZ -- the whole wafer surface can manufacture an almost defect-free silicon single crystal, without decreasing glow in defects, such as FPD of the silicon single crystal manufactured by law, L-SEPD, and COP, and reducing most productivity at a comparatively early pull-up rate. And the oxygen density distribution within a wafer side is also improved.

[Translation done.]

* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1] It is the approximate account Fig. of the crystal-pulling equipment of this invention by the CZ process.
[Drawing 2] It is the approximate account Fig. of the conventional crystal-pulling equipment by the CZ process.
[Drawing 3] It is the explanatory view in which having pulled up the crystal growth theory of this invention with the temperature gradient near the solid-liquid interface during the crystal (growth), and having shown it in relation with a rate as compared with the conventional method.
[Drawing 4] It is the explanatory view in which having pulled up the crystal growth theory of this invention with the temperature gradient near the solid-liquid interface during the crystal (growth), and having shown it with relation with a rate.
[Drawing 5] It is the explanatory view [/ near the solid-liquid interface of this invention] having shown the temperature gradient measuring point.
[Drawing 6] It is the partial expansion explanatory view of drawing 1 having shown the heat insulator arrangement near the solid-liquid interface of the crystal-pulling equipment of this invention.
[Drawing 7] It is the explanatory view in which having pulled up the crystal growth theory of this invention with the temperature gradient near the solid-liquid interface during the crystal (growth), and having shown drawing 3 by more concrete data in relation with a rate as compared with the conventional method.
[Drawing 8] It is the explanatory view in which having pulled up the crystal growth theory of this invention with the temperature gradient near the solid-liquid interface during the crystal (growth), and having shown drawing 4 by more concrete data by relation with a rate.
[Drawing 9] It is the explanatory view having shown the crystal growth theory of this invention during the crystal on the cross section near the solid-liquid interface.
(a): Field distribution by the conventional method.
(b): The whole surface N field by this invention is shown.
[Drawing 10] It is drawing which looked at the situation of change of the crystal defect in the crystal growth direction in an example.
(a): Example 1 (CZ process),
(b): Example 2 (MCZ law).
[Drawing 11] It is the oxygen density measurement result Fig. of an example 3 and the example of a comparison.
(a): The example of a comparison,
(b): Example 3.
[Drawing 12] It is the approximate account Fig. of the crystal-pulling equipment of this invention by the MCZ method.
[Description of Notations]
1 [-- A solid-liquid interface, 5 / -- Seed crystal,] -- A growth single crystal, 2 -- Silicon melt, 3 -- The surface of hot water, 4 6 [-- Up surrounding heat insulator,] -- A seed chuck, 7 -- A cable, 8 -- A solid-liquid interface heat insulator, 9 10 -- The clearance between the surface of hot water and a solid-liquid interface heat insulator lower limit, 30 -- Crystal-pulling equipment, 31 [-- A heater,

35 / -- A heat insulator, 36 / -- A cooling system, 37 / -- A rectification cylinder, 38 / -- A magnet, 39 / -- N field, 40 / -- A V-rich field 41 / -- An I-rich field 42 / -- An OSF field, 43 / -- Growth interface configuration.] -- A pull-up room, 32 -- A crucible, 33 -- A crucible maintenance shaft, 34

[Translation done.]

* NOTICES *

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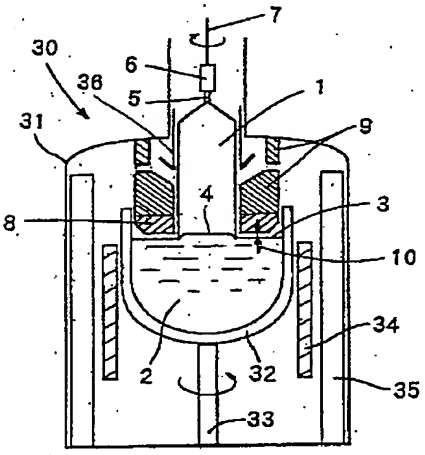
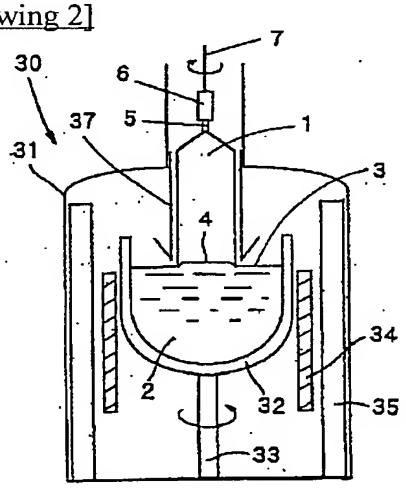
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DRAWINGS

[Drawing 1]



[Drawing 2]



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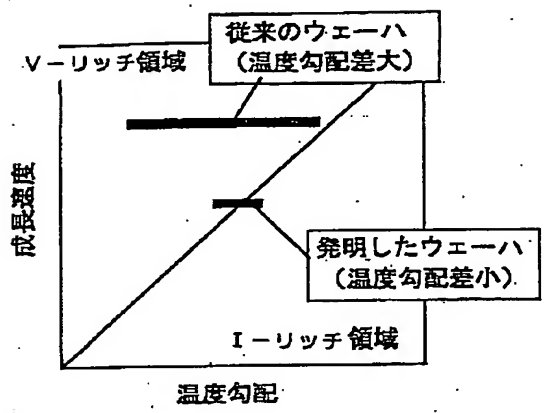

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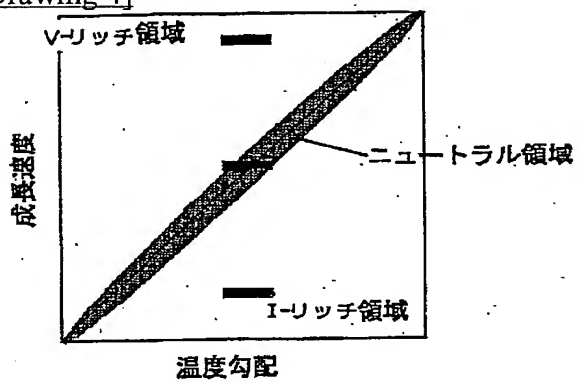

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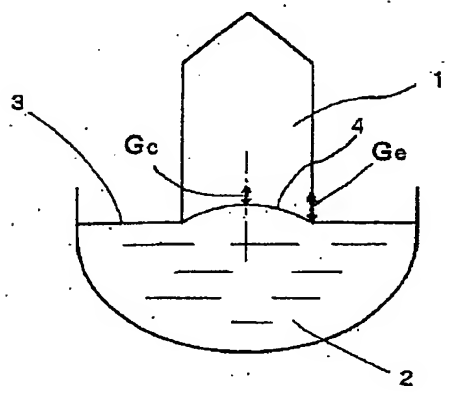

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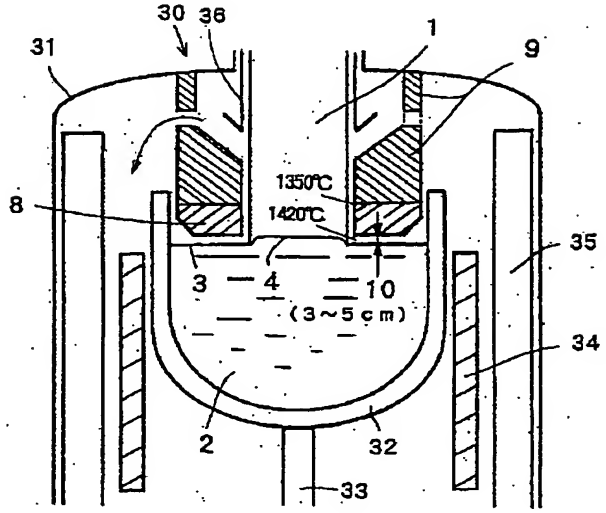
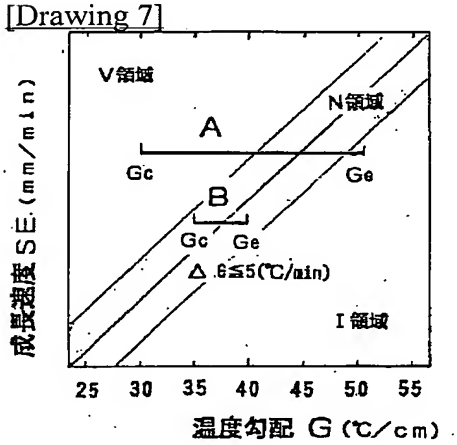
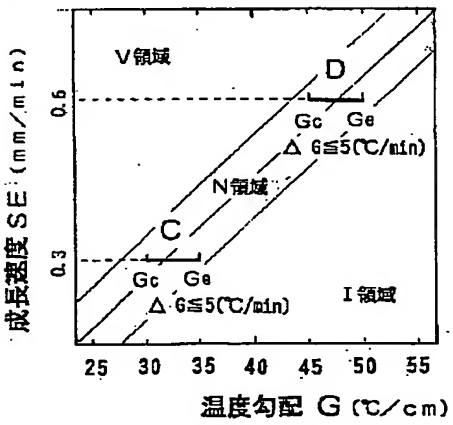


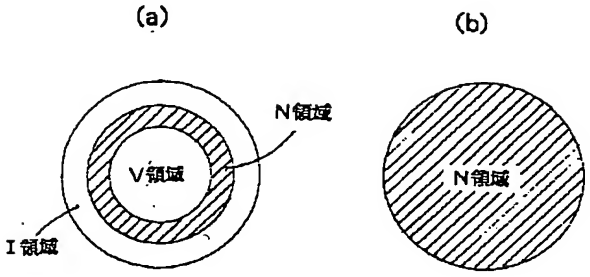
[Drawing 7]



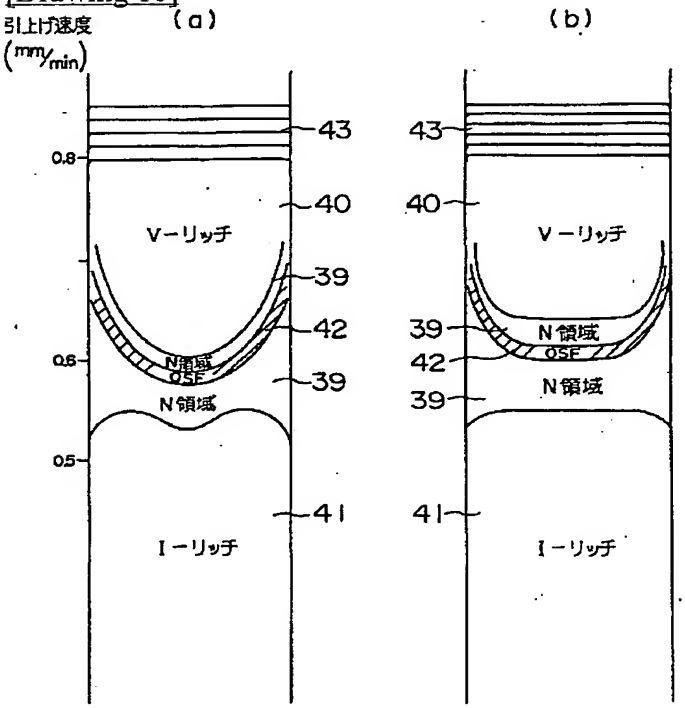
[Drawing 8]



[Drawing 9]

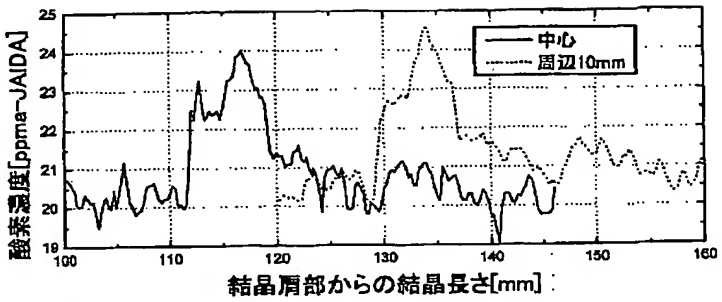


[Drawing 10]

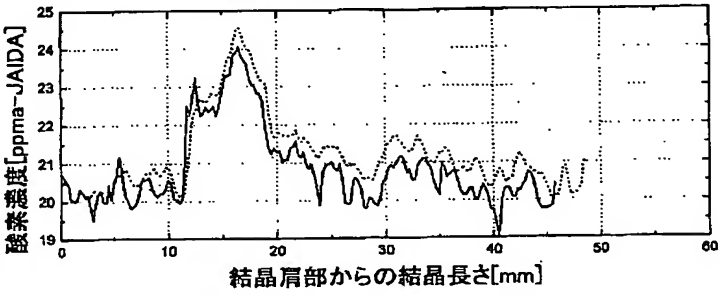


[Drawing 11]

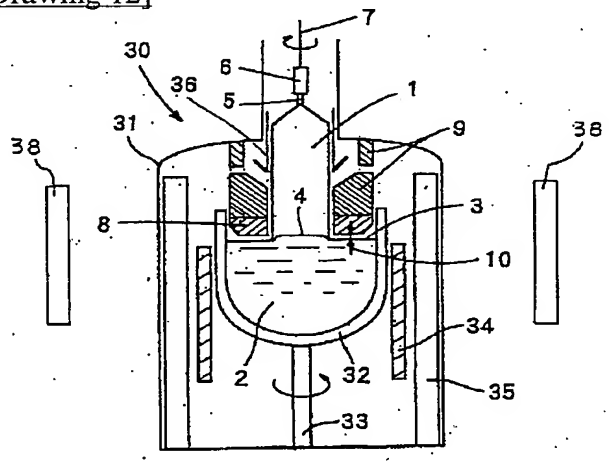
(a)



(b)



[Drawing 12]



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(54) PRODUCTION OF AND PRODUCTION UNIT FOR SILICON SINGLE CRYSTAL WITH FEW CRYSTAL DEFECT, AND SILICON SINGLE CRYSTAL AND SILICON WAFER PRODUCED THEREBY

(57)Abstract:
PROBLEM TO BE SOLVED: To obtain a silicon single crystal and a silicon wafer with extremely low defect density throughout the crystal by CZ process while maintaining their high productivity, along with improving the in-plane oxygen concentration distribution of the wafer.
SOLUTION: This method for producing a silicon single crystal by Czochralski(CZ) process comprises pulling up a silicon single crystal being in growth so that the contour of the solid-liquid interface in the crystal comes within ±5 mm relative to the average of the solid-liquid interface except the periphery 5 mm; wherein for the value of temperature gradient G (temperature change/ axial crystal length) [C/cm] in the proximity of the above interface from 1,420° C to 1,350° C or from the melting point of silicon to 1,400° C, in-oven temperature is controlled so that ΔG comes within 5° C/cm {ΔG= (Ge-Gc); Gc is the temperature gradient in the central portion of the crystal [° C/cm]; Ge is the temperature gradient in the peripheral portion of the crystal [° C/cm]}.

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最終頁に続く

(54)【発明の名称】　結晶欠陥が少ないシリコン単結晶の製造方法、製造装置並びにこの方法、装置で製造されたシリコン単結晶とシリコンウエーハ

(57)【要約】

【課題】　結晶全面に亙って極低欠陥密度であるＣＺ法によるシリコン単結晶及びウエーハを、高生産性を維持しながら得るとともに、合わせてシリコンウエーハの面内酸素濃度分布をも改善する。

【解決手段】　ＣＺ法によってシリコン単結晶を製造する場合において、育成されるシリコン単結晶が結晶成長時に、結晶中の固液界面の形状が、周辺５ｍｍを除いて固液界面の平均値に対し±５ｍｍ以内となるように引き上げるシリコン単結晶の製造方法、およびＣＺ法によってシリコン単結晶を製造する場合において、育成されるシリコン単結晶が結晶成長時に、結晶中の固液界面近傍の１４２０℃から１３５０℃の間の温度勾配またはシリコンの融点から１４００℃の間の温度勾配Ｇ（温度変化量／結晶軸方向長さ）［℃／ｃｍ］の値を、結晶中心部分の温度勾配Ｇｃ［℃／ｃｍ］と結晶周辺部分の温度勾配Ｇｅ［℃／ｃｍ］との差△Ｇ＝（Ｇｅ－Ｇｃ）で表した時、△Ｇが５℃／ｃｍ以内となるように炉内温度を制御するシリコン単結晶の製造方法。

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【特許請求の範囲】

【請求項１】　チョクラルスキー法によってシリコン単結晶を製造する場合において、育成されるシリコン単結晶が結晶成長時に、結晶中の固液界面の形状が、周辺５ｍｍを除いて固液界面の平均値に対し±５ｍｍ以内となるように引き上げることを特徴とするシリコン単結晶の製造方法。

【請求項２】　チョクラルスキー法によってシリコン単結晶を製造する場合において、育成されるシリコン単結晶が結晶成長時に、結晶中の固液界面近傍の１４２０℃から１３５０℃の間の温度勾配またはシリコンの融点から１４００℃の間の温度勾配Ｇ（温度変化量／結晶軸方向長さ）［℃／ｃｍ］の値を、結晶中心部分の温度勾配Ｇｃ［℃／ｃｍ］と結晶周辺部分の温度勾配Ｇｅ［℃／ｃｍ］との差△Ｇ＝（Ｇｅ－Ｇｃ）で表した時、△Ｇが５℃／ｃｍ以内となるように炉内温度を制御することを特徴とするシリコン単結晶の製造方法。

【請求項３】　磁場を印加したチョクラルスキー法によってシリコン単結晶を製造する場合において、育成されるシリコン単結晶が結晶成長時に、結晶中の固液界面近傍の１４２０℃から１３５０℃の間の温度勾配またはシリコンの融点から１４００℃の間の温度勾配Ｇ（温度変化量／結晶軸方向長さ）［℃／ｃｍ］の値を、結晶中心部分の温度勾配Ｇｃ［℃／ｃｍ］と結晶周辺部分の温度勾配Ｇｅ［℃／ｃｍ］との差△Ｇ＝（Ｇｅ－Ｇｃ）で表した時、△Ｇが５℃／ｃｍ以内となるように炉内温度を制御することを特徴とするシリコン単結晶の製造方法。

【請求項４】　前記印加される磁場を水平磁場とすることを特徴とする請求項３記載のシリコン単結晶の製造方法。

【請求項５】　印加する磁場の強度を２０００Ｇ以上とすることを特徴とする請求項３又は請求項４記載のシリコン単結晶の製造方法。

【請求項６】　前記結晶中の１３００℃から１０００℃までの温度域の結晶の長さが８ｃｍ以下となるように制御することを特徴とする、請求項１ないし請求項５のいずれか１項に記載したシリコン単結晶の製造方法。

【請求項７】　前記結晶中の１３００℃から１０００℃までの温度域を通過する時間が８０分以下となるように制御することを特徴とする、請求項１ないし請求項６のいずれか１項に記載したシリコン単結晶の製造方法。

【請求項８】　前記結晶中の固液界面近傍の１４２０℃から１３５０℃の間の温度勾配またはシリコンの融点から１４００℃の間の温度勾配Ｇと引上げ速度を調節して、結晶全面の引上げ状態をベイカンシー・リッチ領域とインタースティシアル・リッチ領域との境界近辺に合わせ、結晶の全面を欠陥濃度の偏りの少ないニュートラルな領域において引上げを行うようにすることを特徴とする、請求項１ないし請求項７のいずれか１項に記載したシリコン単結晶の製造方法。

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【請求項９】　チョクラルスキー法によってシリコン単結晶を製造する装置において、育成されるシリコン単結晶が結晶成長時に、結晶中の固液界面近傍の１４２０℃から１３５０℃の間の温度勾配またはシリコンの融点から１４００℃の間の温度勾配Ｇ（温度変化量／結晶軸方向長さ）［℃／ｃｍ］の値を、結晶中心部分の温度勾配Ｇｃ［℃／ｃｍ］と結晶周辺部分の温度勾配Ｇｅ［℃／ｃｍ］との差△Ｇ＝（Ｇｅ－Ｇｃ）で表した時、△Ｇが５℃／ｃｍ以内となるように炉内温度を形成することを特徴とする、シリコン単結晶の製造装置。

【請求項１０】　磁場を印加したチョクラルスキー法によってシリコン単結晶を製造する装置において、育成されるシリコン単結晶が結晶成長時に、結晶中の固液界面近傍の１４２０℃から１３５０℃の間の温度勾配またはシリコンの融点から１４００℃の間の温度勾配Ｇ（温度変化量／結晶軸方向長さ）［℃／ｃｍ］の値を、結晶中心部分の温度勾配Ｇｃ［℃／ｃｍ］と結晶周辺部分の温度勾配Ｇｅ［℃／ｃｍ］との差△Ｇ＝（Ｇｅ－Ｇｃ）で表した時、△Ｇが５℃／ｃｍ以内となるように炉内温度を形成することを特徴とする、シリコン単結晶の製造装置。

【請求項１１】　チョクラルスキー法によってシリコン単結晶を製造する装置において、シリコン溶融液の湯面直上にシリコン単結晶を囲繞するように固液界面断熱材を配置し、シリコン単結晶を囲繞した固液界面断熱材の下端と湯面との間の隙間を３～５ｃｍとすることを特徴とする、請求項９又は請求項１０に記載したシリコン単結晶の製造装置。

【請求項１２】　請求項１ないし請求項８のいずれか１項に記載した方法によって製造されたシリコン単結晶。

【請求項１３】　請求項９ないし請求項１１のいずれか１項に記載した装置によって製造されたシリコン単結晶。

【請求項１４】　チョクラルスキー法により育成されたシリコン単結晶であって、成長方向に垂直な方向の酸素濃度分布が、５％以下であることを特徴とするシリコン単結晶。

【請求項１５】　ＦＰＤ密度が１００ケ／ｃｍ^2 以下であり、かつサイズが１０μｍ以上のＳＥＰＤ密度が１０ケ／ｃｍ^2 以下であることを特徴とするシリコンウエーハ。

【請求項１６】　酸素濃度の面内分布が、５％以下であることを特徴とする請求項１５のシリコンウエーハ。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は、結晶欠陥が少なく、酸素濃度分布が均一なシリコン単結晶の製造方法、製造装置、並びにこの方法、装置で製造されたシリコン単結晶とシリコンウエーハに関するものである。

【０００２】

【従来の技術】近年は、半導体回路の高集積化に伴う素子の微細化に伴い、その基板となるチョクラルスキー法（ＣＺ法）で作製されたシリコン単結晶に対する品質要求が高まってきている。特に、ＦＰＤ、ＬＳＴＤ、ＣＯＰ等のグローンイン（Ｇｒｏｗｎ－ｉｎ）欠陥と呼ばれる欠陥の密度とサイズの低減が要求されている。

【０００３】これらの欠陥を説明するに当たって、先ず、シリコン単結晶に取り込まれるベイカンシイ（Ｖａｃａｎｃｙ、以下Ｖと略記することがある）と呼ばれる空孔型の点欠陥と、インタースティシアルーシリコン（Ｉｎｔｅｒｓｔｉｔｉａｌ－Ｓｉ、以下Ｉと略記することがある）と呼ばれる格子間型シリコン点欠陥のそれぞれの取り込まれる濃度を決定する因子について、一般的に知られていることを説明する。

【０００４】シリコン単結晶において、Ｖ領域とは、Ｖａｃａｎｃｙ、つまりシリコン原子の不足から発生する凹部、穴のようなものが多い領域であり、Ｉ領域とは、シリコン原子が余分に存在することにより発生する転位や余分なシリコン原子の塊が多い領域のことであり、そしてＶ領域とＩ領域の間には、原子の不足や余分が無い（少ない）ニュートラル（Ｎｅｕｔｒａｌ、以下Ｎと略記することがある）領域が存在していることになる。そして、前記グローンイン欠陥（ＦＰＤ、ＬＳＴＤ、ＣＯＰ）というのは、あくまでもＶやＩが過飽和な状態の時に発生するものであり、多少の原子の偏りがあっても、飽和以下であれば、欠陥としては存在しないことが判ってきた。

【０００５】この両点欠陥の濃度は、ＣＺ法における結晶の引上げ速度と結晶中の固液界面近傍の温度勾配Ｇとの関係（図４参照）から決まり、Ｖ領域とＩ領域との境界近辺にＯＳＦと呼ばれる別種の欠陥の存在が確認されているというのが通説となっている（Ｅｒｉｃｈ Ｄｏｒｎｂｅｒｇｅｒ ａｎｄ Ｗｉｌｆｒｅｄ ｖｏｎＡｍｍｏｎ，Ｊ．Ｅｌｅｃｔｒｏｃｈｅｍ．Ｓｏｃ．，Ｖｏｌ．１４３，Ｎｏ．５，Ｍａｙ １９９６．、Ｔ．Ａｂｅ，Ｈ．Ｈａｒａｄａ，Ｊ．Ｃｈｉｋａｗａ，Ｐａｐｅｒ ｐｒｅｓｅｎｔｅｄ ａｔ ＩＣＤＳ－１２ Ａｍｓｔｅｒｄａｍ，Ａｕｇｕｓｔ３１～Ｓｅｐｔｅｍｂｅｒ３，１９８２参照）。

【０００６】これらの中で、従来の引上げ法は、製造コストやＯＳＦは存在しないという考えから、結晶成長速度の速い領域であるＶ－リッチと呼ばれる領域内での製造が多く行われ、この領域の結晶内の欠陥を、引上げ中の熱履歴等を制御して低減化を図っていた。例えば、１１５０～１０８０℃の通過時間を長くし、ＦＰＤ等、Ｖの集まりと思われる欠陥の密度を低減し、デバイス特性に近い評価ができる酸化膜耐圧特性を改善する、といった方法である。しかし、このような方法等、引上げ中の熱履歴（いずれかの温度帯域の通過時間）を制御するやり方では、欠陥の密度は減らせても欠陥サイズが拡大し、トータルな欠陥の総体積は減少されていないことが最近判ってきた。

【０００７】そこで、最近では製造コストは増加するが、品質改善のために引上げ速度を下げるか、可能な限り結晶中の固液界面近傍の温度勾配を大きくして、結晶の一部又は全面にＩ－リッチと呼ばれるＦＰＤ、ＬＳＴＤ、ＣＯＰ等の欠陥があまり観察されない領域を使用する試みがなされつつある。ところが、最近の研究でＩ－リッチ領域の中でもＶとＩの境界領域から離れたようなところでは、過剰な格子間シリコンが集まって形成する転位ループと考えられている大きいＳＥＰＤと呼ばれる欠陥が存在することが判った。この大きいＳＥＰＤ（以下、Ｌ－ＳＥＰＤという）は、Ｖ領域に存在するＦＰＤ、ＬＳＴＤ、ＣＯＰ等よりもデバイスの特性に悪影響を与える恐れがある。

【０００８】一方、半導体デバイスの高集積化は、シリコンウエーハ面内の均一性の要求も求めており、中でも酸素濃度分布は、作製されるデバイス歩留りに直接影響を及ぼすため、特にウエーハ面内で均一に分布していることが望まれる。チョクラルスキー法で製造されるシリコンウエーハ面内の酸素濃度分布が悪化する原因としては、シリコン融液の対流、ガス雰囲気条件あるいは結晶回転、ルツボの回転等、種々のものが考えられるが、特には成長する結晶棒の外側の周辺部と、内側の中心部とでは冷却されやすさが異なるために、結晶成長界面（固液界面）が平らにならないことが挙げられる。

【０００９】すなわち、通常チョクラルスキー法における結晶成長界面は、内側が冷却されにくいことから成長が遅れ、上側に凸形状のものとなり、これをスライスして得たウエーハの面内では、成長時期が異なるために、成長縞を有することになる。その結果、ウエーハ面内では、結晶成長方向での酸素濃度の変動に従った分布を有することになる。

【００１０】しかし、従来はこのような界面形状に基づく酸素濃度の変動、分布は、チョクラルスキー法で棒状の単結晶棒を引上げ製造する方法においては、必然的に起るものとされ、やむを得ないものと考えられていた。したがって、程度の差こそあれ、界面形状に基づく変動、分布は有する上で、前記結晶回転等の他のファクタを制御することによって、出来るだけウエーハ面内の酸素濃度分布を改善しようとした。

【００１１】

【発明が解決しようとする課題】本発明は、このような問題点に鑑みなされたもので、Ｖ－リッチ領域およびＩ－リッチ領域のいずれも存在しない、結晶全面に亙って極低欠陥密度であるＣＺ法によるシリコン単結晶及びウエーハを、高生産性を維持しながら得るとともに、合わせてシリコンウエーハの面内酸素濃度分布をも改善することを目的とする。

【００１２】

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【課題を解決するための手段】本発明は、前記目的を達成するために為されたもので、本発明の請求項１に記載した発明は、チョクラルスキー法によってシリコン単結晶を製造する場合において、育成されるシリコン単結晶が結晶成長時に、結晶中の固液界面の形状が、周辺５ｍｍを除いて固液界面の平均値に対し±５ｍｍ以内となるように引き上げることを特徴とするシリコン単結晶の製造方法である。

【００１３】このように、結晶成長界面（固液界面）形状を、周辺５ｍｍを除いて固液界面の平均値に対し±５ｍｍ以内となるように平坦化して結晶を引き上げれば、欠陥の多いＶ－リッチ領域とＩ－リッチ領域とが混在しない中性領域で、結晶を引き上げることが可能となるとともに、ウエーハ面内の酸素濃度分布を著しく改善することができる。周辺５ｍｍを除いたのは、この領域では固液界面の形状が急激に変化しており安定しないからである。

【００１４】また、本発明の請求項２に記載した発明は、ＣＺ法によってシリコン単結晶を製造する場合において、育成されるシリコン単結晶が結晶成長時に、結晶中の固液界面近傍の１４２０℃から１３５０℃の間の温度勾配またはシリコンの融点から１４００℃の間の温度勾配Ｇ（温度変化量／結晶軸方向長さ）［℃／ｃｍ］の値を、結晶中心部分の温度勾配Ｇｃ［℃／ｃｍ］と結晶周辺部分の温度勾配Ｇｅ［℃／ｃｍ］との差△Ｇ＝（Ｇｅ－Ｇｃ）で表した時、△Ｇが５℃／ｃｍ以内となるように炉内温度を制御するようにした。

【００１５】このように、結晶成長時に、いわゆるホットゾーン（以下、ＨＺという）の調整により、すなわち、結晶中心部分の温度勾配Ｇｃ［℃／ｃｍ］と結晶周辺部分の温度勾配Ｇｅ［℃／ｃｍ］との差△Ｇ＝（Ｇｅ－Ｇｃ）で表した時、△Ｇが５℃／ｃｍ以内となるように炉内温度を制御すれば、Ｖ－リッチ領域とＩ－リッチ領域の間に存在するＮ領域のみでの引上げを可能とし、その引上げ速度を決めることができるので、欠陥の多いＶ－リッチ領域とＩ－リッチ領域とが混在しない中性領域で、結晶全面に亙って極低欠陥密度であるＣＺ法によるシリコン単結晶及びウエーハを、安定状態で高生産性を維持しながら製造することができる。そして、このように温度勾配の差△Ｇを５℃／ｃｍ以内とすることによって、結晶育成時の固液界面形状を、周辺５ｍｍを除いて固液界面の平均値に対し±５ｍｍ以内に平坦化することができ、酸素濃度分布を改善することが出来る。この場合、結晶中の固液界面近傍の温度勾配Ｇ（温度変化量／結晶軸方向長さ）［℃／ｃｍ］としては、１４２０℃から１３５０℃の間の温度勾配を用いても、シリコンの融点から１４００℃の間の温度勾配を用いてもよいが、シリコンの融点から１４００℃の間の温度勾配を用いた方がより正確な制御をすることができる。

【００１６】次に、本発明の請求項３に記載した発明

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は、磁場を印加したチョクラルスキー法によってシリコン単結晶を製造する場合において、育成されるシリコン単結晶が結晶成長時に、結晶中の固液界面近傍の１４２０℃から１３５０℃の間の温度勾配またはシリコンの融点から１４００℃の間の温度勾配Ｇ（温度変化量／結晶軸方向長さ）［℃／ｃｍ］の値を、結晶中心部分の温度勾配Ｇｃ［℃／ｃｍ］と結晶周辺部分の温度勾配Ｇｅ［℃／ｃｍ］との差△Ｇ＝（Ｇｅ－Ｇｃ）で表した時、△Ｇが５℃／ｃｍ以内となるように炉内温度を制御することを特徴とするシリコン単結晶の製造方法である。

【００１７】このように、磁場を印加したチョクラルスキー法において△Ｇが５℃／ｃｍ以内となるようにして結晶を引き上げれば、Ｎ領域が広がり、その制御範囲が広くなるために、より簡単に結晶欠陥がほとんど無いシリコン単結晶、シリコンウエーハを製造することが出来る。

【００１８】この場合、請求項４に記載したように、印加される磁場を水平磁場とし、また請求項５に記載したように、印加する磁場の強度を２０００Ｇ以上とするのが好ましい。シリコン融液の対流を抑制してＮ領域を広げると共に、固液界面形状を平坦化するには、水平磁場の方が縦磁場あるいはカスプ磁場より好ましいし、２０００Ｇ未満では、磁場印加効果が少ないからである。

【００１９】そして、本発明においては、結晶中の１３００℃から１０００℃までの温度域の結晶長さが８ｃｍ以下となるように制御し（請求項６）、結晶中の１３００℃から１０００℃までの温度域を通過する時間が８０分以下となるように制御することが好ましい（請求項７）。

【００２０】これは、前記結晶中の固液界面より上部の固体結晶部分の温度勾配或は引上げ速度を規定した引上げ条件であり、こうすることによって、温度勾配Ｇの絶対値そのものが大きくなり、高い引上げ速度によってもニュートラル領域で引き上げることができる。

【００２１】結晶中の１３００℃から１０００℃までの温度域の結晶長さが８ｃｍを越える場合は、温度勾配Ｇの絶対値が小さくなり、極端に遅い引上速度でしか結晶全面に亙って極低欠陥密度であるシリコン単結晶およびウエーハを得ることが出来なくなる。同様に、結晶中の１３００℃から１０００℃までの温度域を通過する時間が８０分を超えた場合でも温度勾配Ｇの絶対値が小さくなり、結晶全面に亙って極低欠陥密度であるシリコン単結晶およびウエーハを得ようとすると引上速度を遅くせざるを得ず、安定状態で高生産性を維持することが困難となる。

【００２２】また、本発明の請求項８に記載した発明は、結晶中の固液界面近傍の１４２０℃から１３５０℃の間の温度勾配またはシリコンの融点から１４００℃の間の温度勾配Ｇと引上げ速度を調節して、結晶全面の引上げ状態をＶ・リッチ領域とＩ・リッチ領域との境界近

辺に合わせ、結晶の全面を欠陥濃度の偏りの少ないニュートラル（Ｎ、中性）な領域において行うようにするというものである。

【００２３】従来のＮ領域という概念を全く考慮しない方法で結晶を引上げると、温度勾配の差△Ｇ＝（Ｇｅ－Ｇｃ）が大きくなってしまい結晶面内でＮ領域だけを作ることは出来なかったが、前記したように△Ｇ＝（Ｇｅ－Ｇｃ）≦５［℃／ｃｍ］に制御するとともに、引上げ速度を調整することにより結晶全面にＮ領域が出来るようになった。こうすることによってＶ、Ｉのいずれの欠陥の発生も少なく、結晶の全面において欠陥濃度が低く、その偏りの少ないニュートラルな領域が得られ、安定した品質が確保される。

【００２４】次に、本発明の請求項９に記載した発明は、チョクラルスキー法によってシリコン単結晶を製造する装置において、育成されるシリコン単結晶が結晶成長時に、結晶中の固液界面近傍の１４２０℃から１３５０℃の間の温度勾配またはシリコンの融点から１４００℃の間の温度勾配Ｇ（温度変化量／結晶軸方向長さ）［℃／ｃｍ］の値を、結晶中心部分の温度勾配Ｇｃ［℃／ｃｍ］と結晶周辺部分の温度勾配Ｇｅ［℃／ｃｍ］との差△Ｇ＝（Ｇｅ－Ｇｃ）で表した時、△Ｇが５℃／ｃｍ以内となるように炉内温度を形成することを特徴とする、シリコン単結晶の製造装置である。

【００２５】また、本発明の請求項１０に記載した発明は、磁場を印加したチョクラルスキー法によってシリコン単結晶を製造する装置において、育成されるシリコン単結晶が結晶成長時に、結晶中の固液界面近傍の１４２０℃から１３５０℃の間の温度勾配またはシリコンの融点から１４００℃の間の温度勾配Ｇ（温度変化量／結晶軸方向長さ）［℃／ｃｍ］の値を、結晶中心部分の温度勾配Ｇｃ［℃／ｃｍ］と結晶周辺部分の温度勾配Ｇｅ［℃／ｃｍ］との差△Ｇ＝（Ｇｅ－Ｇｃ）で表した時、△Ｇが５℃／ｃｍ以内となるように炉内温度を形成することを特徴とする、シリコン単結晶の製造装置である。

【００２６】そして、本発明の請求項１１に記載した発明は、請求項９または請求項１０に記載の装置であって、チョクラルスキー法によってシリコン単結晶を製造する装置において、シリコン溶融液の湯面直上にシリコン単結晶を囲繞するように固液界面断熱材を配置し、シリコン単結晶を囲繞した固液界面断熱材の下端と湯面との間の隙間を３～５ｃｍとすることを特徴とする、シリコン単結晶の製造装置である。

【００２７】このように、△Ｇが５℃／ｃｍ以内となる装置、特に磁場を印加した装置であれば、結晶欠陥の無いＮ領域のみで結晶を引き上げることが可能となる。例えば、ＨＺを取り巻く固液界面断熱材下端を湯面から３～５ｃｍの隙間を設けて設置した構造にすれば、ヒータの輻射熱が固液界面に十分当たり、固液界面近傍の温度勾配の差△Ｇ＝（Ｇｅ－Ｇｃ）を５℃／ｃｍ以下に制御

することが可能なシリコン単結晶の製造装置を形成することが可能となり、結果的には、結晶欠陥の極めて少ない品質のシリコン単結晶およびシリコンウエーハを安定して製造することができる。

【００２８】この固液界面断熱材下端と湯面との隙間が３ｃｍ未満の場合は、ヒータ等からの熱輻射が不十分となり、結果として固液界面近傍の温度勾配の差△Ｇ＝（Ｇｅ－Ｇｃ）が５℃／ｃｍを越える値となり、欠陥の多いＶ－リッチ領域とＩ－リッチ領域とが混在しない中性領域で、結晶全面に亙って極低欠陥密度であるシリコン単結晶およびウエーハを作ることができなくなる。一方、この固液界面断熱材下端と湯面との隙間が５ｃｍを超える場合は、温度勾配Ｇそのものの値が小さくなり、必然的に結晶全面に亙って極低欠陥密度であるシリコン単結晶およびウエーハを得るためには引上げ速度を極端に遅くする必要が生じ、これでは安定状態で高生産性を維持することは難しい。

【００２９】さらに、本発明の請求項１２、請求項１３に記載した発明は、前記請求項１ないし請求項８のいずれか１項に記載した方法あるいは請求項９ないし請求項１１のいずれか１項に記載した装置によって製造されたシリコン単結晶である。

【００３０】このように、前記請求項１ないし請求項８のいずれか１項に記載した方法および請求項９ないし請求項１１のいずれか１項に記載した装置によってシリコン単結晶を製造すれば、Ｎ領域で安定的に結晶を引上げられるため、ＦＰＤ、ＬＳＴＤ、ＣＯＰ、Ｌ－ＳＥＰＤ等の結晶欠陥が極めて少ないとともに、ウエーハ面内の酸素濃度分布を改善したシリコン単結晶を製造することができる。

【００３１】次に、本発明の請求項１４に記載した発明は、チョクラルスキー法により育成されたシリコン単結晶であって、成長方向に垂直な方向の酸素濃度分布が、５％以下であることを特徴とするシリコン単結晶である。このように、本発明では、△Ｇを５℃／ｃｍ以内とし、固液界面形状を平坦化するので、酸素濃度分布が均一なシリコン単結晶を得ることができる。特に、本発明では、１本の単結晶棒のほとんどがその成長方向に垂直な方向（スライス後のウエーハ面内方向）で均一なものとなる。

【００３２】そして、本発明の請求項１５に記載した発明は、ＦＰＤ密度が１００ケ／ｃｍ^2 以下であり、かつサイズが１０μｍ以上のＳＥＰＤ密度が１０ケ／ｃｍ^2 以下であることを特徴とするシリコンウエーハである。

【００３３】このように、本発明によって製造されたシリコン単結晶から作製されるシリコンウエーハは、ＦＰＤやＬＳＴＤ、ＣＯＰ、Ｌ－ＳＥＰＤ等のグローンイン欠陥が極めて少ないものであり、極めて有用なシリコンウエーハとすることができる。

【００３４】しかも、請求項１６に記載したように、本

発明のシリコンウエーハは、結晶欠陥が少ないとともに、酸素濃度の面内分布も、５％以下とすることができる。ここで、酸素濃度の面内分布の表示の仕方は種々のものがあるが、本発明では、面内で測定された最大値と最小値の差を最大値で割った値、あるいは面内で測定された最大値と最小値の差を平均値で割った値のいずれでも５％以下とすることができる。

【００３５】以下、本発明につき詳細に説明するが、本発明はこれらに限定されるものではない。説明に先立ち各用語につき予め解説しておく。

１）ＦＰＤ（Ｆｌｏｗ Ｐａｔｔｅｒｎ Ｄｅｆｅｃｔ）とは、成長後のシリコン単結晶棒からウェーハを切り出し、表面の歪み層を弗酸と硝酸の混合液でエッチングして取り除いた後、$K_2Cr_2O_7$と弗酸と水の混合液で表面をエッチング（Ｓｅｃｃｏエッチング）することによりピットおよびさざ波模様が生じる。このさざ波模様をＦＰＤと称し、ウェーハ面内のＦＰＤ密度が高いほど酸化膜耐圧の不良が増える（特開平４－１９２３４５号公報参照）。

【００３６】２）ＳＥＰＤ（Ｓｅｃｃｏ Ｅｔｃｈ Ｐｉｔ Ｄｅｆｅｃｔ）とは、ＦＰＤと同一のＳｅｃｃｏエッチングを施した時に、流れ模様（ｆｌｏｗ ｐａｔｔｅｒｎ）を伴うものをＦＰＤと呼び、流れ模様を伴わないものをＳＥＰＤと呼ぶ。この中で１０μｍ以上の大きいＳＥＰＤは転位クラスターに起因すると考えられ、デバイスに転位クラスターが存在する場合、この転位を通じて電流がリークし、Ｐ－Ｎジャンクションとしての機能を果たさなくなる。

【００３７】３）ＬＳＴＤ（Ｌａｓｅｒ Ｓｃａｔｔｅｒｉｎｇ ＴｏｍｏｇｒａｐｈｙＤｅｆｅｃｔ）とは、成長後のシリコン単結晶棒からウエーハを切り出し、表面の歪み層を弗酸と硝酸の混合液でエッチングして取り除いた後、ウエーハを劈開する。この劈開面より赤外光を入射し、ウエーハ表面から出た光を検出することでウエーハ内に存在する欠陥による散乱光を検出することができる。ここで観察される散乱体については学会等ですでに報告があり、酸素析出物とみなされている（Ｊ．Ｊ．Ａ．Ｐ．　Ｖｏｌ．３２，Ｐ３６７９，１９９３参照）。また、最近の研究では、八面体のボイド（穴）であるという結果も報告されている。

【００３８】４）ＣＯＰ（Ｃｒｙｓｔａｌ Ｏｒｉｇｉｎａｔｅｄ Ｐａｒｔｉｃｌｅ）とは、ウエーハの中心部の酸化膜耐圧を劣化させる原因となる欠陥で、Ｓｅｃｃｏエッチではピットになる欠陥が、アンモニア過酸化水素水洗浄（$NH_4OH:H_2O_2:H_2O=1:1\sim2:5\sim7$の混合液による洗浄）では選択エッチ液として働き、ＣＯＰになる。このピットの直径は１μｍ以下で光散乱法で調べる。

【００３９】本発明者らは、ＣＺ法によるシリコン単結晶成長に関し、Ｖ領域とＩ領域の境界近辺について、詳細に調査したところ、この境界近辺の極く狭い領域にＦＰＤ、ＬＳＴＤ、ＣＯＰの数が著しく少なく、大きなＳＥＰＤも存在しないニュートラルな領域があることを発見した。

【００４０】そこで、このニュートラルな領域をウエーハ全面に広げることができれば、点欠陥を大幅に減らせると発想した。図４に示したように、前記した成長（引上げ）速度と温度勾配の関係の中で、結晶のウエーハ面内では、引上げ速度はほぼ一定であるから、面内の点欠陥の濃度分布を決定する主な因子は温度勾配である。つまり、ウエーハ面内で、軸方向の温度勾配に差があることが問題で、この差を減らすことが出来れば、ウエーハ面内の点欠陥の濃度差も減らせることを見出した。

【００４１】しかも、このようにウエーハ面内で軸方向の温度勾配の差をなくせば、引上げシリコン単結晶の固液界面を平坦化することができ、ウエーハ面内酸素濃度分布の改善も出来ることがわかった。

【００４２】ここで、通常の引上げ方法の場合における、図５に示したような、結晶中心部の温度勾配Ｇｃと結晶周辺部分の温度勾配Ｇｅとの差を調査したところ、（Ｇｅ－Ｇｃ）で少なくとも１５℃／ｃｍあり、特に、温度勾配Ｇの絶対値を大きくした場合には、（Ｇｅ－Ｇｃ）も広がり、時には４０℃／ｃｍも差があることが確認された。

【００４３】このように結晶中心部と周辺部分との温度勾配に差があると、図５のように固液界面（結晶成長界面）４の形状が平坦とはならず、上側に凸形状となる。そして、上記のように温度勾配の差△Ｇが１５℃／ｃｍもあると、中心部の固液界面が周辺５ｍｍを除いて固液界面の平均値に対し±５ｍｍを越えるようになる。したがって、このような従来のチョクラルスキー法による結晶棒からスライスして得たウエーハの面内では、成長時期が異なることに起因する成長縞を有することになり、結晶成長方向での酸素濃度の変動に従った分布を持つことになる。

【００４４】そこで、温度勾配Ｇの差を減らす方法を、例えば、ＦＥＭＡＧと呼ばれる総合伝熱解析ソフト（Ｆ．Ｄｕｐｒｅｔ，Ｐ．Ｎｉｃｏｄｅｍｅ，Ｙ．Ｒｙｃｋｍａｎｓ，Ｐ．Ｗｏｕｔｅｒｓ，ａｎｄ Ｍ．Ｊ．Ｃｒｏｃｈｅｔ，Ｉｎｔ．Ｊ．Ｈｅａｔ Ｍａｓｓ Ｔｒａｎｓｆｅｒ，３３，１８４９（１９９０））を使用して鋭意調査したところ、図６に示したように、結晶の高温域である１４２０～１３５０℃またはシリコンの融点から１４００℃の範囲を断熱材で保温し、また固液界面近傍には融液からの輻射熱を直接当てるようにし、一方これより低温の部分を出来るだけ冷却すればよいことが判った。

【００４５】具体的には、シリコン溶融液の湯面直上にシリコン単結晶を囲繞するように固液界面断熱材を配置し、シリコン単結晶を囲繞した固液界面断熱材の下端と

湯面との間に３～５ｃｍの隙間を設けて設置すれば、ヒータの輻射熱が固液界面に十分当たり、結晶の成長速度と温度勾配との関係を示した図３のような温度勾配の差△Ｇ＝（Ｇｅ－Ｇｃ）が５℃／ｃｍ以下となる熱分布が形成され、Ｖ、Ｉ欠陥の少ないホットゾーンが存在することが明らかになった。

【００４６】よって、シリコン単結晶の引上げ方法において、前記のような温度勾配、すなわち、結晶中の固液界面近傍の１４２０℃（シリコンの融点）から１３５０℃の間の温度勾配またはシリコンの融点から１４００℃の間の温度勾配Ｇ（温度変化量／結晶軸方向長さ）［℃／ｃｍ］の値を、結晶中心部分の温度勾配Ｇｃ［℃／ｃｍ］と結晶周辺部分の温度勾配Ｇｅ［℃／ｃｍ］との差△Ｇ＝（Ｇｅ－Ｇｃ）で表した時、△Ｇが５℃／ｃｍ以内となるように炉内温度をＨＺにより調整すれば、Ｖリッチ領域とＩリッチ領域の間に存在するＮ領域の結晶の引上げ速度を決めることができ、ウエーハ全面がニュートラルになり、点欠陥はほとんど見られなくなった（図４参照）。

【００４７】図４の上部に見られるように△Ｇ≦５℃／ｃｍのＨＺにおいても、成長速度が速や過ぎるとＶリッチ領域の結晶となり、遅過ぎると下部に見られるようにＩリッチ領域の結晶になってしまい、適切な成長速度を選択することで、図４の真ん中に見られるように全面Ｎリッチ領域の単結晶が得られる。

【００４８】この温度勾配の制御と結晶領域の関係をより具体的に説明すると、図７に示したように、従来のＣＺ法では、例えば、Ｇｃ＝３０℃／ｃｍ、Ｇｅ＝５０℃／ｃｍとすると、△Ｇ＝Ｇｅ－Ｇｃ＝２０℃／ｃｍのＨＺであり、例えば図７のＡの位置で比較的早い成長速度で育成すると、この状態での結晶領域は、図９（ａ）に示したように単結晶横断面で見ると、中心部と周辺部は、夫々結晶欠陥の多いＶ領域、Ｉ領域であり、欠陥の少ないＮ領域はこれらの中間部に円環状に存在するに過ぎない。

【００４９】これに対して、本発明の方法では、例えば、図７のＢの位置に見られるように、Ｇｃ＝３５℃／ｃｍ、Ｇｅ＝４０℃／ｃｍとすると、△Ｇ＝Ｇｅ－Ｇｃ＝５℃／ｃｍのＨＺであり、比較的遅い成長速度で育成しており、この状態での結晶領域は、図９（ｂ）に示したように単結晶横断面で見ると、全面が欠陥の少ないＮ領域となった。

【００５０】しかし、この状態では成長速度が遅いので、図８に示したように、Ｃの位置からＤの位置まで温度勾配の差△Ｇ＝Ｇｅ－Ｇｃ≦５℃／ｃｍを維持したまま、かつ温度勾配Ｇ（ＧｃとＧｅ）の絶対値を大きくしたＨＺにすれば、早い成長速度で全面Ｎ領域を達成出来るようになり、高い生産性を保つことができる。

【００５１】また、この現象を固液界面より上部に位置する既に結晶化した部分の温度勾配で見ると、１３００℃から１０００℃までの温度域の結晶長さが８ｃｍ以下となるように制御することで達成できることが判った。結晶中で８ｃｍよりこの温度域が広がると温度勾配の絶対値は小さくなるので、遅い引上げ速度を選択しなければならず、生産効率を悪化させる結果となる。

【００５２】さらに、この現象を引上げ速度の面からみると、既に結晶化した部分における１３００℃から１０００℃までの温度域を通過する時間が８０分以下となるように制御することが必要である。８０分を越えるような徐冷によっても温度勾配の絶対値は小さくなり、Ｎ領域での結晶を得るためには引上げ速度を遅くしなければならなくなり、生産効率が低下することになる。

【００５３】そして、このように△Ｇを５℃／ｃｍ以内として引き上げると、固液界面形状は、周辺５ｍｍを除いて固液界面の平均値に対し±５ｍｍ以内となり、特には±２．５ｍｍ以内となって、Ｎ領域の結晶を成長しやすくなるとともに、酸素濃度の面内分布も改善されることになる。

【００５４】以上のように、本発明のニュートラル領域での引上げ条件を明らかにしてきたが、これらをまとめてみると、結晶中の固液界面近傍の１４２０℃から１３５０℃の間の温度勾配またはシリコンの融点から１４００℃の間の温度勾配Ｇと引上げ速度を調節して、結晶全面の引上げ状態をベイカンシー（Ｖ）・リッチ領域とインタースティシアル（Ｉ）・リッチ領域との境界近辺に合わせ、結晶の全面を欠陥濃度の偏りの少ないニュートラルな領域において行うようにするということである。そして、これによって酸素濃度の面内分布も著しく改善される。

【００５５】なお、△Ｇを５℃／ｃｍ以内とし、温度勾配の絶対値を所望値とした場合における、結晶の全面をＮ領域で引き上げるための引上げ速度を知るには、例えば△Ｇが５℃／ｃｍ以内となるように単結晶棒を高速で引上げ、Ｖ－リッチ結晶が出来るようにし、その後△Ｇを５℃／ｃｍ以内に維持したまま徐々に成長速度を低下させ、最終的にＩ－リッチ結晶ができる成長速度にまで低下させる。そして、出来た単結晶棒を縦方向に切断して結晶欠陥を調べれば、Ｖ－リッチとＩ－リッチとの間にあるＮ領域の成長速度を知ることが出来る。

【００５６】この場合、シリコン融液に磁場を印加するチョクラルスキー法（ＭＣＺ法）により△Ｇを５℃／ｃｍ以内に保ちつつ結晶を引き上げると、上記Ｎ領域が広がり、Ｎ領域となる引上げ速度の範囲が広がって、容易に単結晶をＮ領域として引き上げることが可能となる。

【００５７】

【発明の実施の形態】以下、本発明の実施形態について、図面を参照しながら詳細に説明する。まず、本発明のＣＺ法による単結晶引上装置の構成例を図１により説明する。図１に示すように、この単結晶引上装置３０は、引上げ室３１と、引上げ室３１中に設けられたルツ

ボ３２と、ルツボ３２の周囲に配置されたヒータ３４と、ルツボ３２を回転させるルツボ保持軸３３及びその回転機構（図示せず）と、シリコンの種子結晶５を保持するシードチャック６と、シードチャック６を引き上げるケーブル７と、ケーブル７を回転又は巻き取る巻取機構（図示せず）を備えて構成されている。ルツボ３２は、その内側のシリコン融液（湯）２を収容する側には石英ルツボが設けられ、その外側には黒鉛ルツボが設けられている。また、ヒータ３４の外側周囲には断熱材３５が配置されている。

【００５８】また、本発明の製造方法に関わる製造条件を設定するために、結晶の固液界面の外周に環状の固液界面断熱材８を設け、その上に上部囲繞断熱材９が配置されている。この固液界面断熱材８は、その下端とシリコン融液２の湯面との間に３～５ｃｍの隙間１０を設けて設置されている。上部囲繞断熱材９は条件によっては使用しないこともある。さらに、冷却ガスを吹き付けたり、輻射熱を遮って単結晶を冷却する筒状の冷却装置３６を設けている。

【００５９】別に、最近では図１２に見られるように引上げ室３１の水平方向の外側に、常伝導あるいは超電導コイル等からなる磁石３８を設置し、シリコン融液２に水平方向あるいは垂直方向等の磁場を印加することによって、融液の対流を抑制し、単結晶の安定成長をはかる、いわゆるＭＣＺ法が用いられることも多い。融液に印加される磁場の方向は、磁石の配置によって簡単に変更することが出来る。例えば、一つのコイルを引上げ室３１を水平方向に取り囲むように配置すれば、融液には垂直方向の磁場（縦磁場）が印加されることになり、二つのコイルを引上げ室３１の水平方向の外側で対向配置すれば、融液には水平方向の磁場（横磁場）が印加されることになる。そして、本発明においても、前述のようにこのＭＣＺ法を用いれば、Ｎ領域が広がり制御範囲が広がって容易にＮ結晶を得ることが出来るようになる。

【００６０】次に、上記図１の単結晶引上装置３０による単結晶育成方法について説明する。　まず、ルツボ３２内でシリコンの高純度多結晶原料を融点（約１４２０℃）以上に加熱して融解する。次に、ケーブル７を巻き出すことにより湯２の表面略中心部に種子結晶５の先端を接触又は浸漬させる。その後、ルツボ保持軸３３を適宜の方向に回転させるとともに、ケーブル７を回転させながら巻き取り種子結晶５を引上げることにより、単結晶育成が開始される。以後、引上速度と温度を適切に調節することにより略円柱形状の単結晶棒１を得ることができる。

【００６１】この場合、本発明では、本発明の目的を達成するために次のような設備を施した。先ず、図６（図１の部分拡大説明図）に示したように、引上げ室３１の湯面上の単結晶１中の液状部分の外周空間において、湯面近傍の結晶の温度が１４２０℃から１３５０℃（あるいはシリコンの融点から１４００℃）までの温度域、いわゆるＨＺに環状の固液界面断熱材８を設け、その上に上部囲繞断熱材９を配置している。この固液界面断熱材８は、その下端とシリコン融液２の湯面３との間に３～５ｃｍの隙間１０を設けて設置されている。上部囲繞断熱材９は条件によっては使用しないこともある。さらに、この断熱材の上部に結晶を冷却する装置、例えば冷却装置３６を設けて、これに上部より冷却ガスを吹きつけて結晶を冷却できるものとし、筒下部に輻射熱反射板を取り付けた構造とすることもある。

【００６２】このように液面の直上の位置に所定の隙間を設けて断熱材を配置し、さらにこの断熱材の上部に結晶を冷却する装置を設けた構造とすることによって、結晶成長界面近傍では輻射熱により保温効果が得られ、結晶の上部ではヒータ等からの輻射熱をカットすることが出来るので、結晶周辺部の温度勾配Ｇｅが小さくなり、結晶中心部の温度勾配Ｇｃとの差も小さくなり、かつ固液界面４もフラットになって、結晶の全面において欠陥濃度の偏りの少ないＮ領域で結晶を引上げることが可能となった。この結晶の冷却装置としては、前記筒状の冷却装置３６とは別に、結晶の周囲を囲繞する空冷ダクトや水冷蛇管等を設けて所望の温度勾配を確保するようにしても良い。

【００６３】また、本発明では、図１２のように引上げ室３１の外側に磁石３８を配置するのが好ましい。この場合、印加される磁場を水平磁場とし、印加する磁場の強度を２０００Ｇ以上、より好ましくは３０００Ｇ以上とする。磁場を印加することによってシリコン融液の対流が抑制されて結晶中のＮ領域が広がる。また、固液界面形状を平坦化するには、水平磁場の方が縦磁場あるいはカスプ磁場等より好ましいし、２０００Ｇ未満では、磁場印加効果が少ないからである。

【００６４】そして本発明では、従来のように極端に引上速度を低下させる必要は全くないものであり、また一定の温度域を徐冷する必要もないため、単結晶の生産性を低下させることなく、品質の改善を図ることができる。しかも、酸素濃度の面内分布も改善されるし、ＭＣＺ法を用いれば、制御範囲が広くなり確実に低欠陥結晶の製造が出来る。

【００６５】図１、図１２の本発明の単結晶引上装置と比較のために従来の装置を図２に示した。基本的な構造については、本発明の引上装置と同じであるが、本発明の固液界面断熱材８、上部囲繞断熱材９や冷却装置３６は装備していない。

【００６６】以上のように、詳細に説明した方法と装置によって製造されたシリコン単結晶は、Ｎ領域で安定的に結晶を引上げられるため、ＦＰＤ、ＬＳＴＤ、ＣＯＰ、Ｌ－ＳＥＰＤ等の結晶欠陥の極めて少ないものとすることができる。また、単結晶のほぼ全体に亙って、結晶成長方向に垂直な方向の酸素濃度分布を、５％以下と

することができる。

【００６７】このようなシリコン単結晶から作製したシリコンウエーハは、その全面において、ＦＰＤ密度が１００ケ／ｃｍ² 以下であり、かつサイズが１０μｍ以上のＳＥＰＤ密度が１０ケ／ｃｍ² 以下であるシリコンウエーハとなり、また、酸素析出熱処理を施すことによって、酸素が析出するような酸素濃度を持つシリコンウエーハに対して、酸素析出熱処理後にＸ線観察した際に、面内の析出のコントラストが一定でストライエーションリングの数が少ないウエーハが得られる。すなわち、成長界面が平坦であるため、ウエーハ面内の均一性がよく、特に面内酸素濃度分布は、５％以下となる。

【００６８】

【実施例】以下、本発明の実施例を示す。

（実施例１）図１に示した引上装置３０で、２０インチ石英ルツボに原料多結晶シリコンを６０Ｋｇチャージし、直径６インチ、方位＜１００＞のシリコン単結晶棒を平均引上速度を１．０～０．４ｍｍ／ｍｉｎに変化させて引上げた（単結晶棒の直胴長さ約８５ｃｍ）。シリコン融液の湯温は１４２０℃、湯面から環状の固液界面断熱材の下端までは、４ｃｍの空間とし、その上に１０ｃｍ高さの環状固液界面断熱材を配置し、湯面から引上げ室天井までの高さをルツボ保持軸を調整して３０ｃｍに設定し、上部囲繞断熱材を配備した。

【００６９】結晶の固液界面近傍のシリコンの融点から１４００℃の間の温度勾配は、Ｇｅ＝４５．０［℃／ｃｍ］、Ｇｃ＝４２．０［℃／ｃｍ］、△Ｇ＝（Ｇｅ－Ｇｃ）＝３．０［℃／ｃｍ］に設定した。　ここで得られた単結晶棒から、ウエーハを切り出し、鏡面加工を施すことによって、シリコン単結晶の鏡面ウエーハを作製した。こうしてできたシリコン単結晶の鏡面ウエーハにつき、前記ＦＰＤ、Ｌ－ＳＥＰＤ欠陥の測定を行った。引上げ速度と欠陥測定結果の関係を表１に示した。また、全く同様にして成長させたシリコン単結晶棒を縦割りにして、成長方向での結晶欠陥の変化の様子を見たのが、図１０（ａ）である。

【００７０】

【表１】

引上げ速度 (mm/min)	温度勾配の差 G=Ge－Gc (℃/cm)	領　域	ＦＰＤ (／cm²)	Ｌ－ＳＥＰＤ (／cm²)
０．８	３．６	Ｖリッチ領域	約１０００	――
０．５５	３．６	Ｎ　領　域	≦２０	――
０．５	３．６	Ｉリッチ領域	≧２０	≧１０

【００７１】表１の結果からわかるように、△Ｇが５℃／ｃｍ以内として、結晶の面内に取り込まれる点欠陥の濃度に差がなくても、引上げ速度が合わなければその結果は、ＶリッチにもＩリッチにもなるので、丁度Ｎ領域に合うような引上げ速度を探索した（図４参照）。その結果、０．５５ｍｍ／ｍｉｎの引上げ速度で引上げた場合に、全面ニュートラルなシリコンウエーハを作製することができた。そして、図１０（ａ）に見られるように、結晶成長界面形状４３は平坦であり、酸素濃度分布が均一なものである。ただし、Ｖ－リッチ領域４０、Ｉ－リッチ領域４１の間にあるＮ領域３９の範囲は比較的狭く、結晶全体をこのＮ領域３９として成長させるには、高精度な制御が必要である。なお、Ｎ領域の間にはＯＳＦ領域４２が見られる。

【００７２】（実施例２）次に、図１２に示した引上装置で、３０００Ｇの水平磁場を融液に印加しつつ結晶を引き上げた。磁場を印加したこと以外は、実施例１と同じ条件とした。すなわち、２０インチ石英ルツボに原料多結晶シリコンを６０Ｋｇチャージし、直径６インチ、方位＜１００＞のシリコン単結晶棒を平均引上速度を１．０～０．４ｍｍ／ｍｉｎに変化させて引上げた。シリコン融液の湯温は１４２０℃、湯面から環状の固液界面断熱材の下端までは、４ｃｍの空間とし、その上に１０ｃｍ高さの環状固液界面断熱材を配置し、湯面から引上げ室天井までの高さをルツボ保持軸を調整して３０ｃｍに設定し、上部囲繞断熱材を配備した。

【００７３】結晶の固液界面近傍のシリコンの融点から１４００℃の間の温度勾配は、Ｇｅ＝４５．０［℃／ｃ

ｍ］、Ｇｃ＝４２．０［℃／ｃｍ］、△Ｇ＝（Ｇｅ－Ｇｃ）＝３．０［℃／ｃｍ］に設定した。　ここで得られた単結晶棒を縦割りにして、成長方向での結晶欠陥の変化の様子を見たのが、図１０（ｂ）である。

【００７４】図１０（ｂ）を見ると、通常ＣＺ法と同様、０．５５～０．５８ｍｍ／ｍｉｎ付近の引上げ速度で引上げた場合に、全面ニュートラルなシリコンウエーハを作製することができることがわかる。また、結晶成長界面形状４３も平坦であり、酸素濃度分布も均一なものとなる。

【００７５】そして、図１０（ａ）の通常ＣＺ法と大きく異なるのは、Ｎ領域が非常に広がり、引上げ速度の制御範囲が著しく広がること、またＶ－リッチ領域、Ｎ領域、Ｉ－リッチ領域の境界も平坦化し、これをスライスしてウエーハとする場合に、同一ウエーハ面内ですべてＮ領域となりやすいものとなることがわかる。

【００７６】このように、磁場を印加すると結晶欠陥の発生する様子が著しく変わる理由の詳細は今のところ不明であるが、磁場を印加することにより融液の対流が安定化されることから、融液中の温度勾配が変化し、結晶中への欠陥の取り込み量そのものが変化するのと、成長界面近傍の結晶中の温度勾配も融液中の温度勾配の変化の影響を受け、安定化し結晶欠陥のない理想的な温度勾配となったためであると考えられる。

【００７７】（実施例３、比較例）次に、固液界面形状が、ウエーハ面内酸素濃度に与えている影響を調べてみた。図１２に示した引上装置で、２０インチ石英ルツボに原料多結晶シリコンを６０Ｋｇチャージし、３０００Ｇの磁場を印加しながら、直径８インチ、方位＜１００＞のシリコン単結晶棒を引上げた。シリコン融液の湯温は１４２０℃、湯面から環状の固液界面断熱材の下端までは、４ｃｍの空間とし、その上に１０ｃｍ高さの環状固液界面断熱材を配置し、湯面から引上げ室天井までの高さをルツボ保持軸を調整して３０ｃｍに設定し、上部囲繞断熱材を配備した。

【００７８】結晶の固液界面近傍のシリコンの融点から１４００℃の間の温度勾配は、Ｇｅ＝３２．６［℃／ｃｍ］、Ｇｃ＝３０．５［℃／ｃｍ］、△Ｇ＝（Ｇｅ－Ｇｃ）＝２．１［℃／ｃｍ］に設定した。得られたシリコン単結晶棒を縦割りにして、結晶中心部と周辺部における成長方向での酸素濃度の変化の様子を見たのが、図１１（ｂ）である（実施例３）。この場合、結晶中の固液界面の形状は、周辺５ｍｍを除いて固液界面の平均値に対し±２ｍｍ以内の上に凸形状でほとんど平坦であった。

【００７９】一方、固液界面断熱材および上部囲繞断熱材を取りはずし、その他の条件は上記と同一にして、３０００Ｇの磁場を印加しつつ、直径８インチのシリコン単結晶棒を育成させた。この時、結晶の固液界面近傍の温度勾配は、Ｇｅ＝６３．５［℃／ｃｍ］、Ｇｃ＝３０．４［℃／ｃｍ］、△Ｇ＝（Ｇｅ－Ｇｃ）＝３３．１［℃／ｃｍ］に設定した。得られたシリコン単結晶棒を縦割りにして、結晶中心部と周辺部における成長方向での酸素濃度の変化の様子を見たのが、図１１（ａ）である（比較例）。この場合、結晶中の固液界面の形状は、周辺５ｍｍを除いて固液界面の平均値に対し±１０ｍｍの上に凸形状であった。

【００８０】図１１を見ると、（ａ）では酸素濃度が、結晶の中心部と周辺部とでは大きな差があり、成長方向で酸素濃度が大きくばらついていることがわかる。そして、中心部での酸素濃度の成長方向の変動と周辺部での酸素濃度の変動とは、約１２～２０ｍｍの位相差でほぼ同様の変動をしていることがわかる。これは、結晶成長界面の凸形状をそのまま反映させたものである。

【００８１】一方、（ｂ）では酸素濃度が、結晶の中心部と周辺部とでほぼ一致しており、中心部での酸素濃度の成長方向の変動と周辺部での酸素濃度の変動とは、約０～３ｍｍの位相差で極めてよく一致していることがわかる。このシリコン単結晶は成長方向でのばらつきはあるものの、成長方向に垂直な方向の酸素濃度分布が極めて良好で、これをスライスしてウエーハとした場合には面内で酸素濃度の分布が極めてよいものとなる。これは、結晶成長界面が平坦化されたことが反映させたものである。

【００８２】なお、本発明は、上記実施形態に限定されるものではない。上記実施形態は、例示であり、本発明の特許請求の範囲に記載された技術的思想と実質的に同一な構成を有し、同様な作用効果を奏するものは、いかなるものであっても本発明の技術的範囲に包含される。

【００８３】例えば、上記実施形態においては、直径６および８インチのシリコン単結晶を育成する場合につき例を挙げて説明したが、本発明はこれには限定されず、Ｎ領域に合致し、かつ結晶の固液界面近傍の結晶中心部と周辺部との温度勾配の差を小さくする引上げ速度に調節すれば、直径８～１６インチあるいはそれ以上のシリコン単結晶にも適用できる。また、本発明で磁場を印加する場合にも、シリコン融液に水平磁場を印加する場合に限られるものではなく、縦磁場、カスプ磁場等を印加するその他のＭＣＺ法にも適用できることは言うまでもない。

【００８４】

【発明の効果】以上説明したように、本発明により、ＣＺ法、ＭＣＺ法によって製造されるシリコン単結晶のＦＰＤ、Ｌ－ＳＥＰＤ、ＣＯＰ等のグローイン欠陥を減少させ、比較的早い引上げ速度で生産性を殆ど低下させることなく、ウエーハ全面がほぼ無欠陥のシリコン単結晶を製造することができる。しかも、ウエーハ面内の酸素濃度分布も改善される。

【図面の簡単な説明】

【図１】ＣＺ法による本発明の単結晶引上装置の概略説

明図である。

【図２】ＣＺ法による従来の単結晶引上装置の概略説明図である。

【図３】本発明の結晶成長理論を、結晶中固液界面近傍の温度勾配と引上げ（成長）速度との関係において、従来法と比較して示した説明図である。

【図４】本発明の結晶成長理論を、結晶中固液界面近傍の温度勾配と引上げ（成長）速度との関係で示した説明図である。

【図５】本発明の固液界面近傍における、温度勾配測定位置を示した説明図である。

【図６】本発明の単結晶引上装置の固液界面近傍の断熱材配置を示した図１の部分拡大説明図である。

【図７】本発明の結晶成長理論を、結晶中固液界面近傍の温度勾配と引上げ（成長）速度との関係において、従来法と比較して図３をより具体的なデータで示した説明図である。

【図８】本発明の結晶成長理論を、結晶中固液界面近傍の温度勾配と引上げ（成長）速度との関係で図４をより具体的なデータで示した説明図である。

【図９】本発明の結晶成長理論を、結晶中固液界面近傍の横断面で示した説明図である。

＊（ａ）：従来法による領域分布。

（ｂ）：本発明による全面Ｎ領域を示す。

【図１０】実施例における結晶成長方向での結晶欠陥の変化の様子を見た図である。

（ａ）：実施例１（ＣＺ法）、

（ｂ）：実施例２（ＭＣＺ法）。

【図１１】実施例３および比較例の酸素濃度測定結果図である。

（ａ）：比較例、

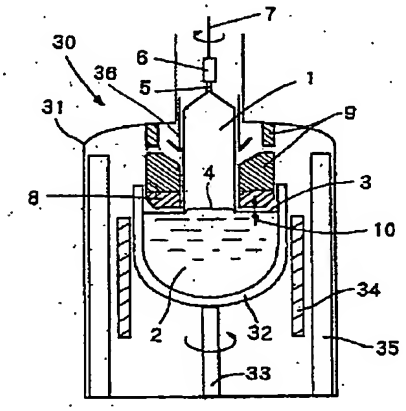
（ｂ）：実施例３。

【図１２】ＭＣＺ法による本発明の単結晶引上装置の概略説明図である。

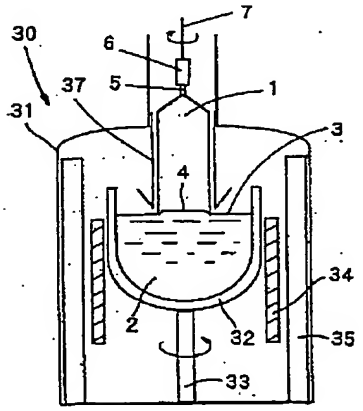
【符号の説明】

１…成長単結晶、２…シリコン融液、３…湯面、４…固液界面、５…種子結晶、６…シードチャック、７…ケーブル、８…固液界面断熱材、９…上部囲繞断熱材、１０…湯面と固液界面断熱材下端との隙間、３０…単結晶引上装置、３１…引上げ室、３２…ルツボ、３３…ルツボ保持軸、３４…ヒータ、３５…断熱材、３６…冷却装置、３７…整流筒、３８…磁石、３９…Ｎ領域、４０…Ｖ－リッチ領域、４１…Ｉ－リッチ領域、４２…ＯＳＦ領域、４３…成長界面形状。

【図１】



【図２】



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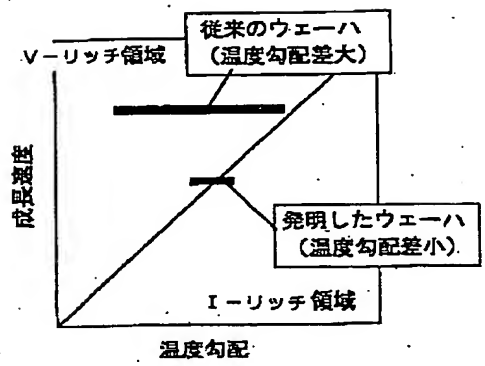

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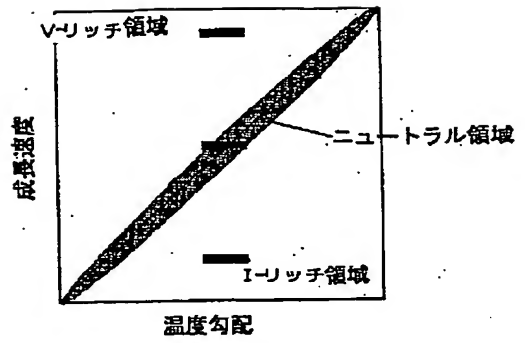

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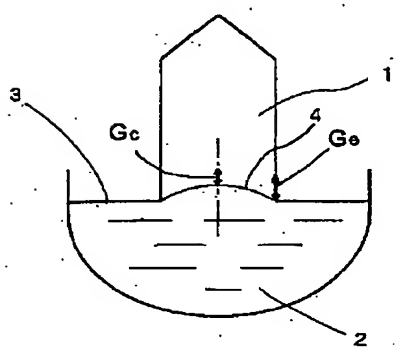

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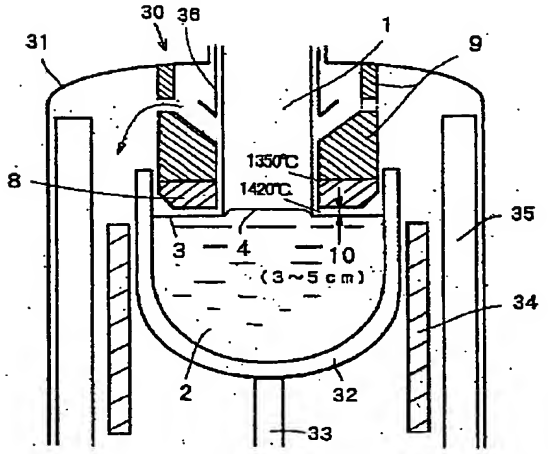

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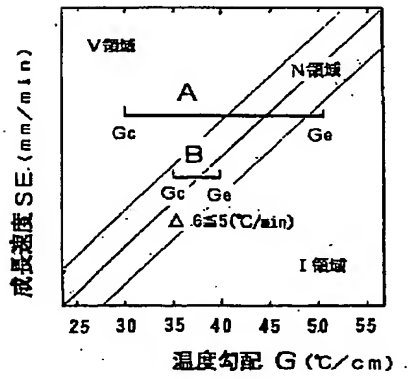

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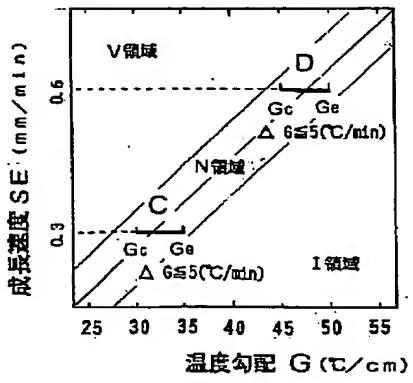

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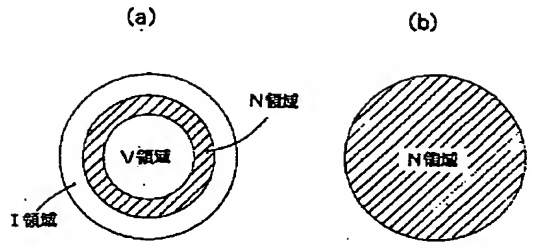
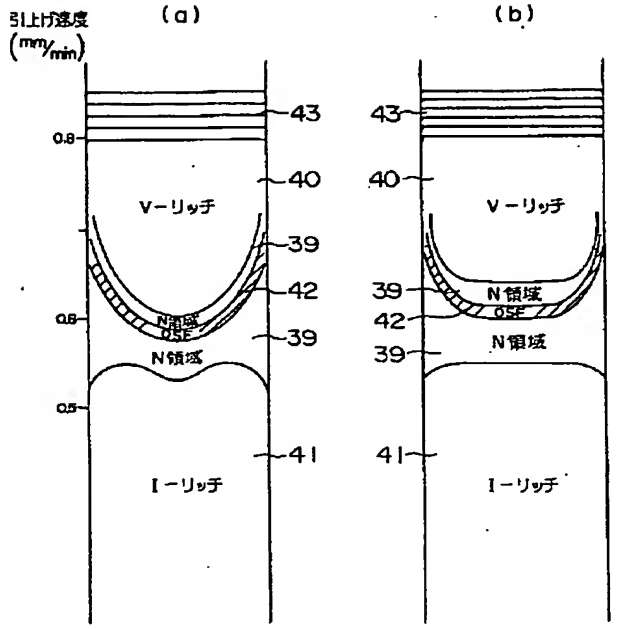
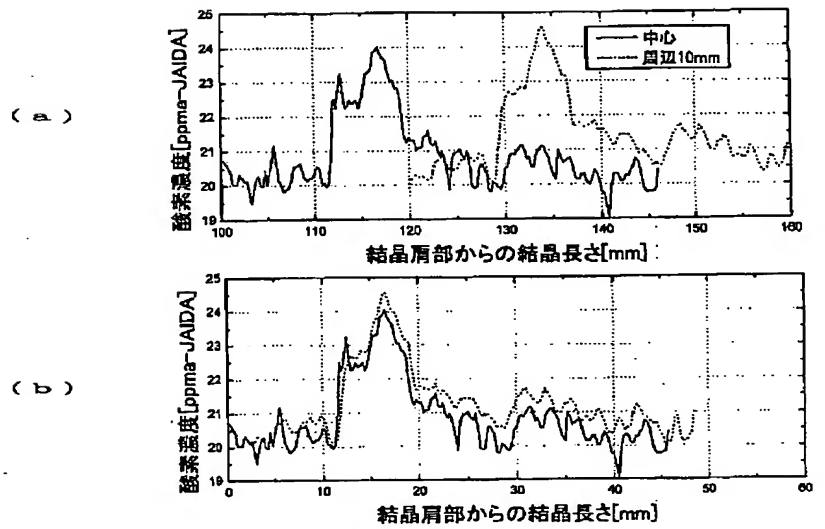

(a)
(b)
N領域
V領域
I領域
N領域

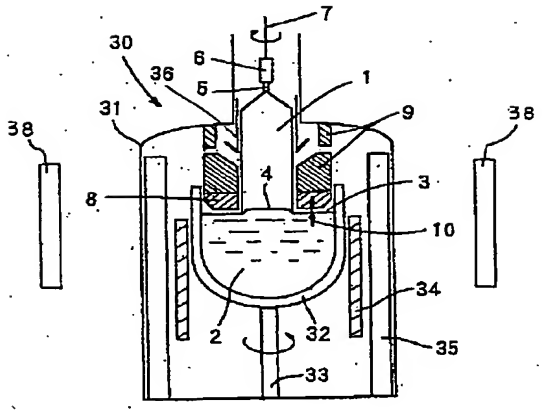
【図１０】


引上げ速度 (mm/min)
(a)
(b)
0.8
0.6
0.5
43
40
39
42
39
41
V－リッチ
N領域
OSF
N領域
I－リッチ
43
40
39
42
39
41
V－リッチ
N領域
OSF
N領域
I－リッチ

【図１１】


(a)
(b)
酸素濃度[ppma-JAIDA]
中心
周辺10mm
結晶肩部からの結晶長さ[mm]
酸素濃度[ppma-JAIDA]
結晶肩部からの結晶長さ[mm]

【図１２】



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